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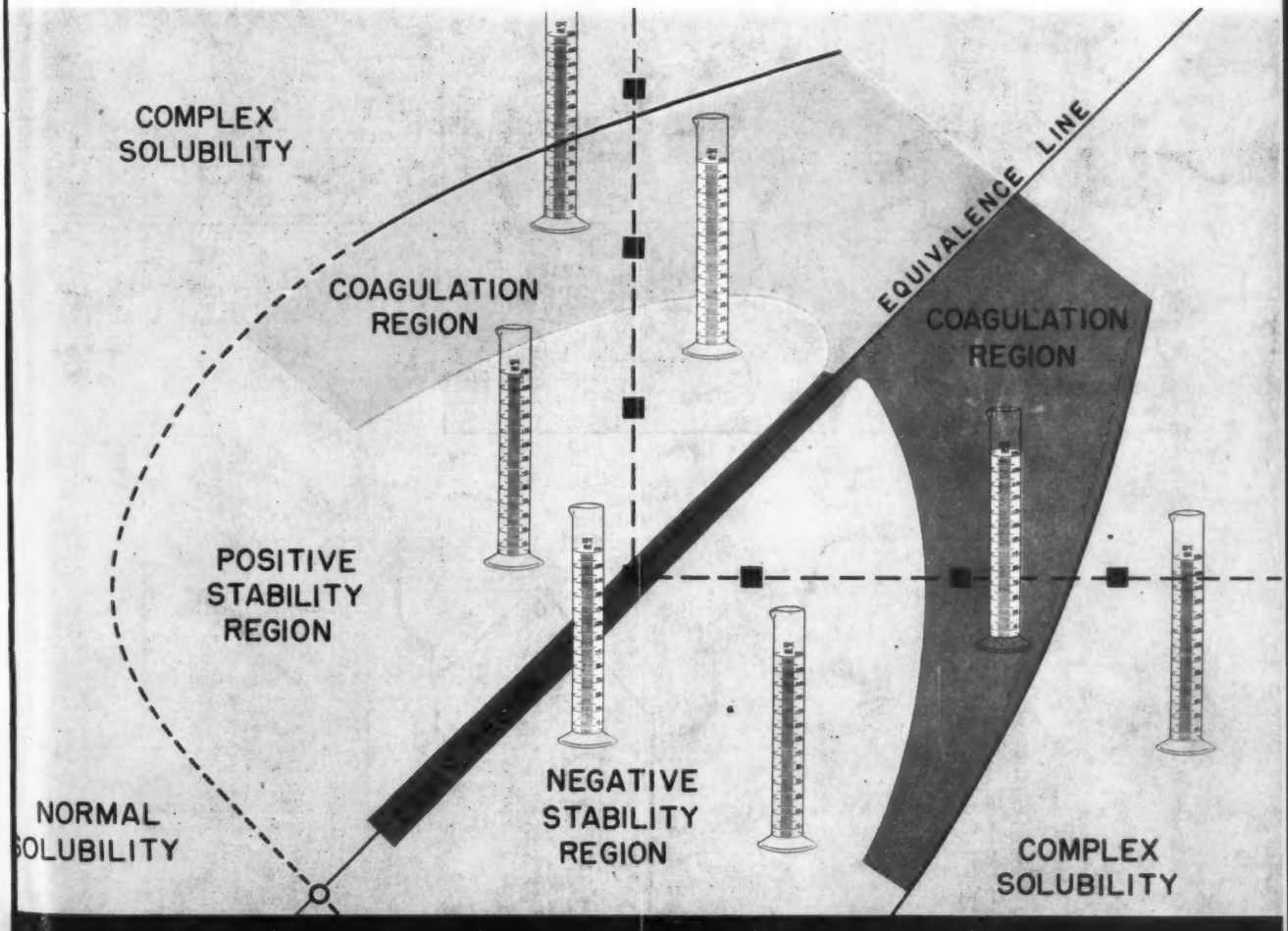

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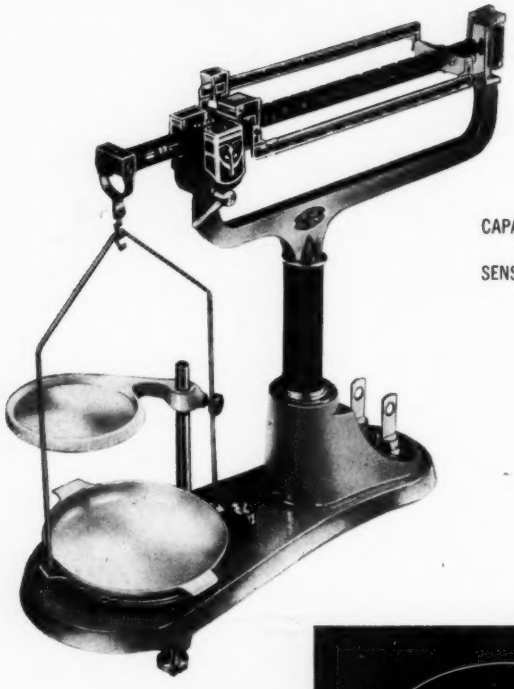

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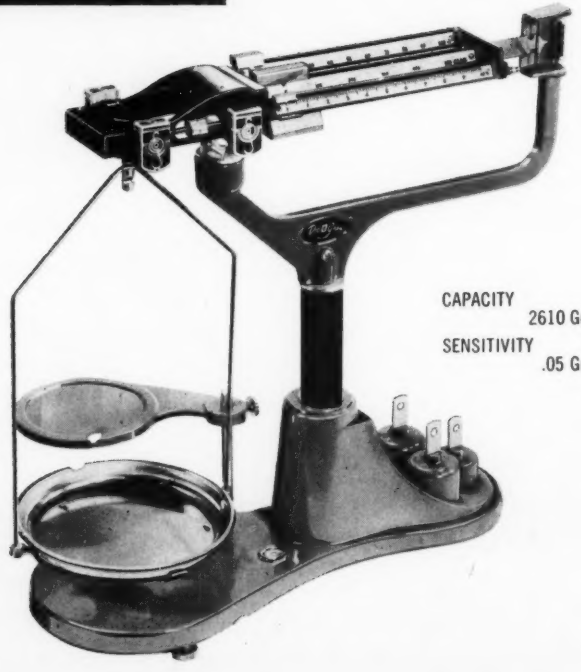

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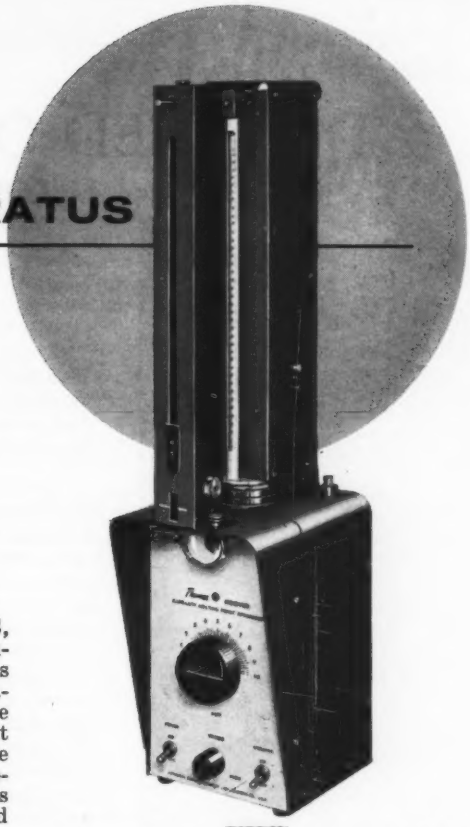

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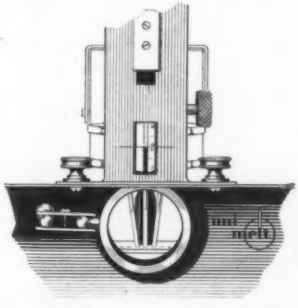

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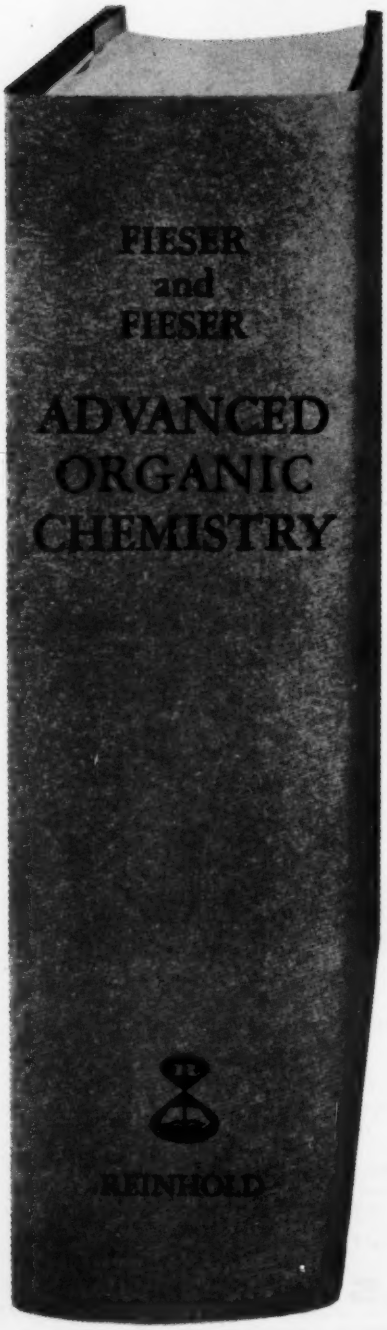

by LOUIS F. FIESER and **MARY FIESER**

Sheldon Emery Professor of Organic Chemistry, Harvard University — *Research Fellow in Chemistry, Harvard University*

Early Fall, 1961, approx. 1,200 pages, 6⅛ x 9¼, $14.00

This new book keeps pace with advances on all fronts in a field that is moving rapidly. The literature coverage is to July 1, 1961. Name-and-date references are cited to 126 papers of 1960 and to 91 papers of 1961. Biographical data are included for 601 chemists, of whom 423 are living.

The Fiesers employ a new plan of organization. The first three chapters include the following topics: Kekulé structures; the covalent bond; the hydrogen bond; Lewis acids and bases; activation energy; transition states and intermediates; orbital theory (CH_4, C_2H_6); structures and names of hydrocarbons, alcohols, amines, acids, carbonyl compounds; conformational stability of derivatives of ethane and of cyclohexane; resonance in the acetate ion; aromatic types and resonance stabilization; inductive effects from pK_a values; and stereochemistry. Presentation of the chemistry of the alkanes, alkenes, etc. is then possible on a comprehensive scale and at a mature level. The authors often illustrate standard reactions with exciting examples taken from a special field: use of three types of OH-protective groups in the synthesis of UDPG; use of the Wittig reaction in the total synthesis of lycopene, vitamin D_3, and quinquephenyl; cortisol, to exemplify the behavior of hydroxyl groups of three types; mechanism of *vic*-glycol cleavage, particularly as evaluated for the case of four rigid stereoisomeric steroid diols; the Velluz synthesis (1960) of gonadal and corticoid hormones.

A key theme of current research involves interpretation and planning of reactions that proceed through unisolable transition states, through intermediates demonstated by trapping techniques (carbenes, benzynes, cyclobutadiene), or through aronium ions of types postulated (phenonium ions) and of types recently isolated (benzeneonium ions). Other topics highlighting recent advances include: ring systems of unconventional size and type, use of alkynes in synthesis, structure and conformation of proteins (molecular biology), peptide synthesis, denaturation and renaturation of the DNA helices.

The book abounds in mechanisms, in details of experimentation, and historical episodes. It is clear, readable, and interesting.

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This series of articles presents a survey of the basic principles, characteristics, and limitations of those instruments which find important applications in chemical work. The emphasis is on commercially available equipment, and approximate prices are quoted to show the order of magnitude of cost of the various types of design and construction.

14. Chromatographic Equipment

Although the term "chromatography" originally referred to a specific technique for the resolution of mixtures of colored substances based on capillary action, usage has led to such a broadening of the application of this term that the original meaning has long been relegated to the limbo of history. To the extent that there is an underlying, unifying principle behind all the techniques that are currently referred to as chromatographic in nature, it is this: *there is a selective distribution of components between two phases, coupled with a force producing relative motion of the phases.*

This simple principle is now exploited in such a broad array of practical techniques that chromatography has developed in about two decades from a sparingly used, esoteric research tool to the most popular and active field in analytical chemistry.

The forms taken by chromatographic equipment are determined by the fundamental mechanisms being utilized for the separation of the components of the samples, and the proper choice and use of such equipment must be predicated upon a thorough grasp of basic principles.

Selective Distribution

Selectivity in the distribution of components between two phases may be achieved in the following ways.

1. Vapor-Liquid Equilibrium. The vapor that is in equilibrium with a two-component liquid is enriched in the more volatile component. That is, the more volatile constituent distributes itself between the two phases so that its concentration is greater in the gas phase than in the liquid phase. The relationship between the concentrations in the two phases can be deduced from simple thermodynamics (using the Duhem-Margules equation), with the result:

$$\frac{p_1}{p_2} = \left(1 - \frac{dP}{dp_2}\right)\frac{x_1}{x_2}$$

where p_1 and p_2 are the partial pressures in the gas phase of components 1 and 2 respectively, P is the total pressure (viz., $p_1 + p_2$), and x_1 and x_2 are the mole fractions of the two components in the liquid phase.

If component 2 is the less volatile substance, increase in its concentration leads to a decrease in the total pressure, and dP/dp_2 will be negative. Hence, p_1/p_2 will be greater than x_1/x_2, and the vapor is relatively richer in component 1 than is the liquid with which it is in equilibrium. This difference in composition of liquid and vapor at the same temperature is illustrated in Figure 1 for an ideal solution.

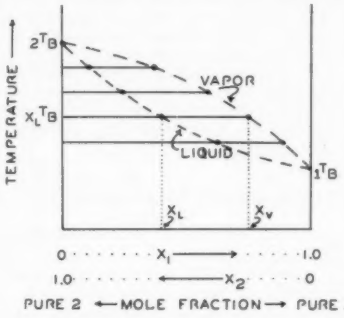

Figure 1. The vapor in equilibrium with a two-component liquid is richer in the more volatile component. $_1T_B$, $_2T_B$ and x_LT_B are the boiling points of pure component 1, pure 2, and solution of composition x_L respectively. The upper dashed curve shows the composition of the vapor phase, the lower curve that of the liquid phase, which can exist in equilibrium with each other at any given temperature.

Considerations similar to the above apply to the selective distribution of the components of a gas mixture between the gas phase and a non-volatile liquid phase in which they can dissolve.

2. Liquid-Liquid Extraction Equilibrium. In the two-phase system formed by a pair of partially miscible liquids, a third component distributes itself between the phases in proportion to its solubility in, or compatibility with, each. This is expressed quantitatively by the Distribution (or, Partition) Law, which in its simplest form is:

$$\frac{C_1}{C_2} = K$$

where C_1 and C_2 are the concentrations of the third component in liquid phases 1 and 2 respectively, and K is the distribution, or partition, coefficient. This equation applies to the case where the state of aggregation of the solute is the same in both phases; when different association or dissociation equilibria are involved in the two phases, more complicated expressions incorporating the respective equilibrium constants apply. Typical distribution isotherms, showing the relationship between the concentrations of solute in the two phases as a function of the solute concentration, are shown in Figure 2.

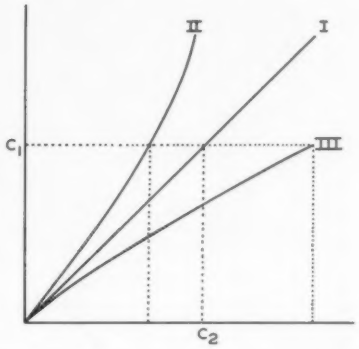

Figure 2. Each curve shows the equilibrium concentrations, C_1 and C_2, of a given solute in two liquid phases, 1 and 2 respectively, as a function of concentration. *I.* A straight line (i.e., constant partition ratio) is characteristic of an equilibrium in which the solute has the same molecular form in both phases. *II.* Positive deviation from constant partition ratio occurs, e.g., when the solute dissociates in phase 1, but not in phase 2. *III.* Negative deviation occurs, e.g., when the solute associates in phase 2, but not in phase 1.

The dissolving of a gas in a liquid is also a partition equilibrium, for the liquid can be thought of as an extractant for the gas, and the latter distributes itself between the two phases. Henry's Law, $c_g = Kp$, where c_g is the concentration of dissolved gas, and p is the gas phase partial pressure, is equivalent to the distribution law given above.

3. Interfacial Adsorption Equilibrium. At the interface between a gas and a solid, a liquid and a solid, two liquids, etc., an adsorption-desorption equilibrium can be established for a solute in the mobile phase. Adsorption of a component is said to occur at an interface if the formation of the interface causes the concentration of the component in its phase to decrease. For example, if activated silica gel is added to a dilute solution of acetic acid in water, the concentration of the acid is observed to decrease, due to adsorption at the liquid-solid interface. Actually, both components of a binary mixture are attracted to and held at the interface, and the net concentration effect observed is due to the competition between these molecules for the limited number of adsorption sites.

If silica gel is added to a dilute solution of water in acetic acid (i.e., the reverse of the situation just described), the concentration of the water is observed to decrease, due to its preferential adsorption. The types of net apparent adsorptions

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occurring with binary mixtures at liquid-solvent interfaces are due to the differences in molecular and electronic structure of the components; the general behaviors commonly observed are illustrated graphically in Figure 3.

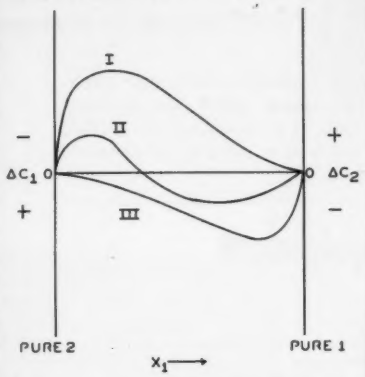

Figure 3. Concentration changes observed in adsorption by a solid phase from a binary liquid. ΔC_1 and ΔC_2 are the concentration changes observed in components 1 and 2 respectively. *I.* Component 1 much more strongly adsorbed than component 2, so that effect of adsorbent is to produce concentration decrease in C_1 for all mixtures. *II.* Both components are adsorbed to comparable extents; component 1 appears to be preferentially adsorbed when 2 is in excess, and vice versa. *III.* Component 2 much more strongly adsorbed than 1.

Chromatographic Development

Given the possibility of achieving an enrichment of one component relative to another by means of one of the distribution mechanisms described above, it then becomes necessary to multiply or cascade the effect a sufficient number of times to achieve analytically useful results. There are two basic approaches to the repetitive application of a selective distribution process to a sample; these may be designated as (*a*) counter-current fractionation, and (*b*) frontal development.

In *counter-current fractionation*, two streams move past each other, traveling in opposite directions, while distribution of components takes place between the streams, as illustrated symbolically in Figure 4. Since there is selectivity in the distribution of the components between the two phases, each component tends to move, and its concentration profile tends to spread out, at a characteristic rate.

This mechanism is involved, and this type of concentration distribution pattern is produced, in a number of well-established techniques; most importantly, counter-current extraction, and elution analysis.

Fractional distillation is also a counter-current distribution process, and is truly a chromatographic technique, involving, as it does, repetitive selective distributions of components in numerous vapor-liquid equilibrations throughout the length of the column. However, the type of distribution produced is different from that just described. In the preceding case, the sample was small compared to the bulk of the two streams between which it was distributed. In a fractional distillation,

(*Continued on page A518*)

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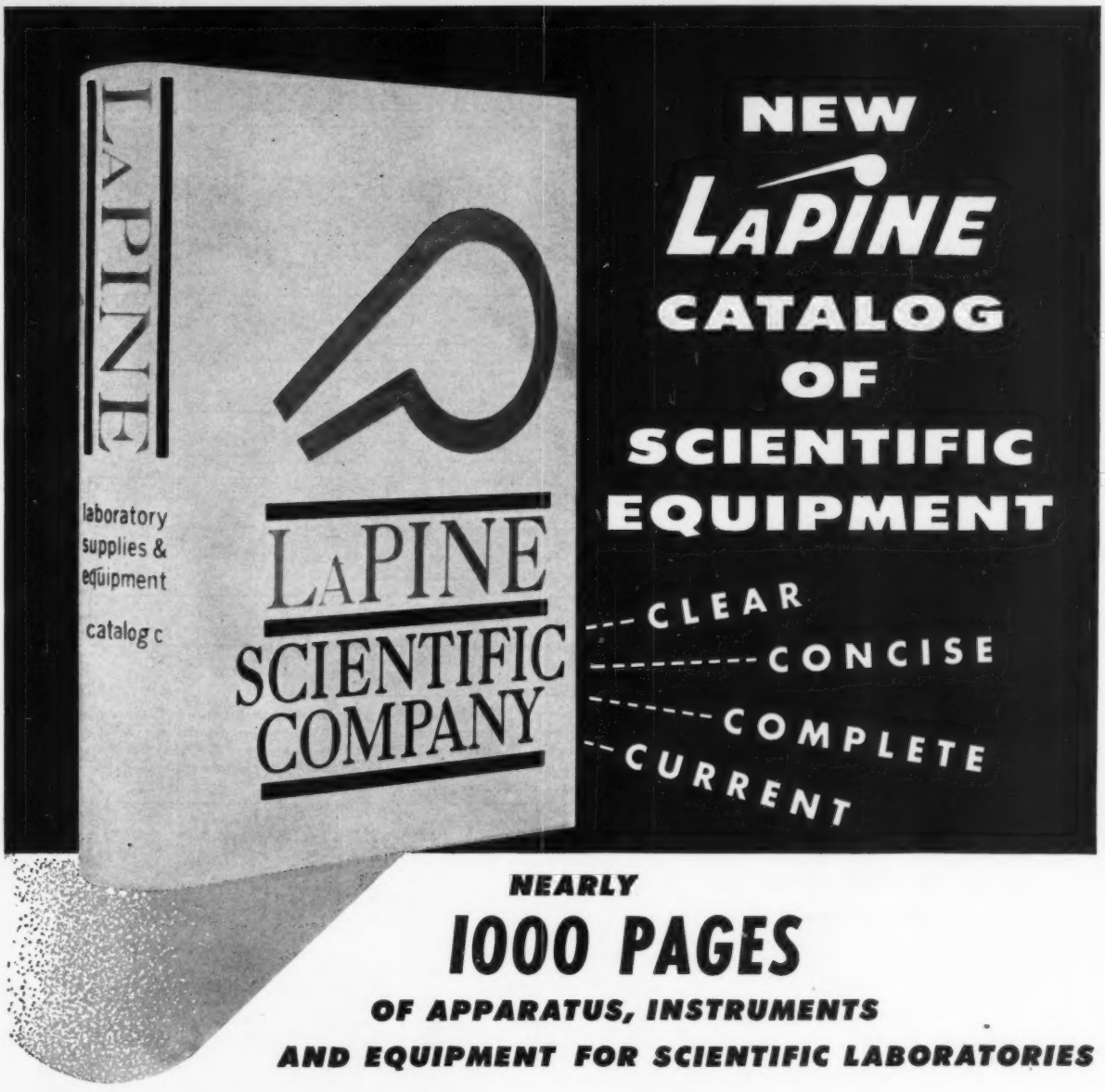

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however, the sample itself also constitutes the two oppositely moving streams, and consequently the distribution has the characteristics illustrated in Figure 5. As the phase diagram in Figure 6 shows (for a simple, non-azeotropic binary system), each liquid on each plate is in equilibrium with vapor that is enriched in the more volatile component. As one moves up the column, the vapor from below is condensed to form a new liquid phase, which in turn produces vapor still richer in the more volatile constituent. The liquid and vapor compositions shown at various places in the diagram of the distilling column in Figure 5 correspond to the points located on the graphs given in Figure 6. The net enrichment of the distillate taken from the top of the column depends upon the number of discrete vapor-liquid equilibrations which have occurred during passage through the length of the column. A single vapor-liquid equilibration is called a *theoretical plate*, and the efficiency of the separation that can be achieved in any case depends upon the number of theoretical plates to which the over-all column is equivalent.

The theoretical plate concept has been extended to all chromatographic processes, and it is not uncommon to describe the efficiency of a chromatographic column in terms of its total theoretical plates, or the HETP, i.e., Height Equivalent to a Theoretical Plate, where the theoretical plate is the equivalent of one distribution or partition equilibration of all components between the two phases involved.

(*Continued on page A520*)

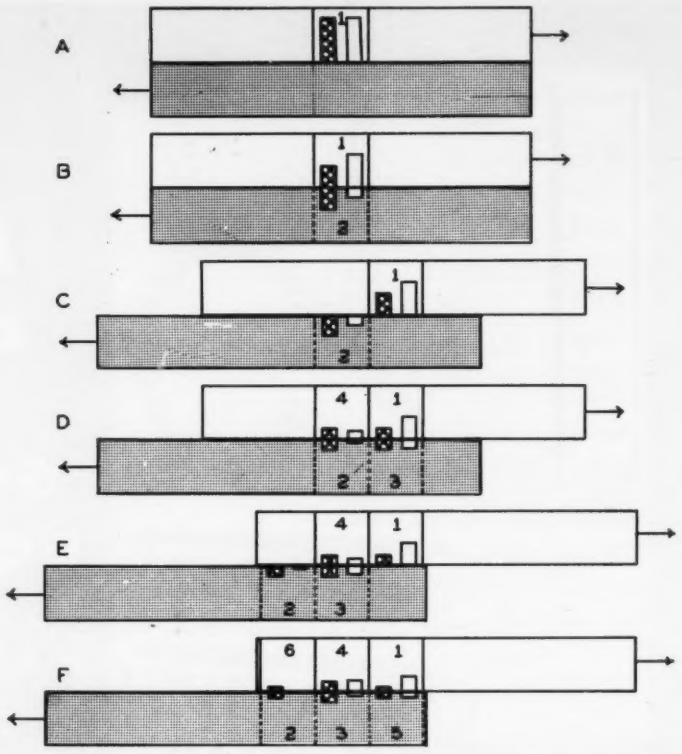

Figure 4. Counter-current fractionation involves selective distribution between two streams moving in opposite directions. Two discontinuous displacements of the streams are shown. *A.* Initial contact. *B.* Partition equilibration is established. *C.* First displacement. *D.* New partition equilibria established. *E.* Displacement occurs again. *F.* New partition equilibria established.

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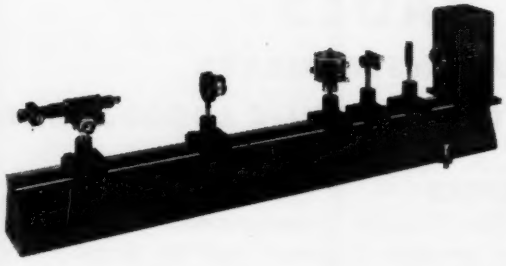

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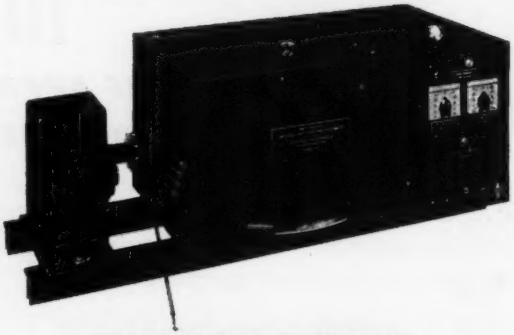

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A form of chromatographic development that is different in fundamental respects from the counter-current fractionation technique is the method of *frontal development*. The principal feature of this method is the use, as one of the phases between which the sample components are to be distributed, of an amount of material which is too small to permit each component of the sample to reach a concentration distribution that is substantially independent of the other constituents. It is typical of an elution analysis that the sample is sufficiently limited in amount, compared to the bulk of adsorbent, so that each component is fully adsorbed, and is moved along by the eluting solvent at a rate determined by its own characteristic properties in the solvent system employed, and not (to a first approximation) by the nature and amounts of the other sample components. However, in a frontal analysis, the total amount of sample is greater than the adsorption capacity of the adsorbent, and the sample components compete with each other for adsorption sites. Hence, the degree of adsorption, and the rate of travel of a given component depend upon the kind and amounts of the other components in the sample.

Consider, for example, a limited amount of an adsorbent through which is passed a large volume of a solution containing equal concentrations of four substances: *A*, *B*, *C*, and *D*. Assume that *A* is more strongly absorbed than *B*, *B* more than *C*, and *C* more than *D*. Figure 7 shows in

(*Continued on page A522*)

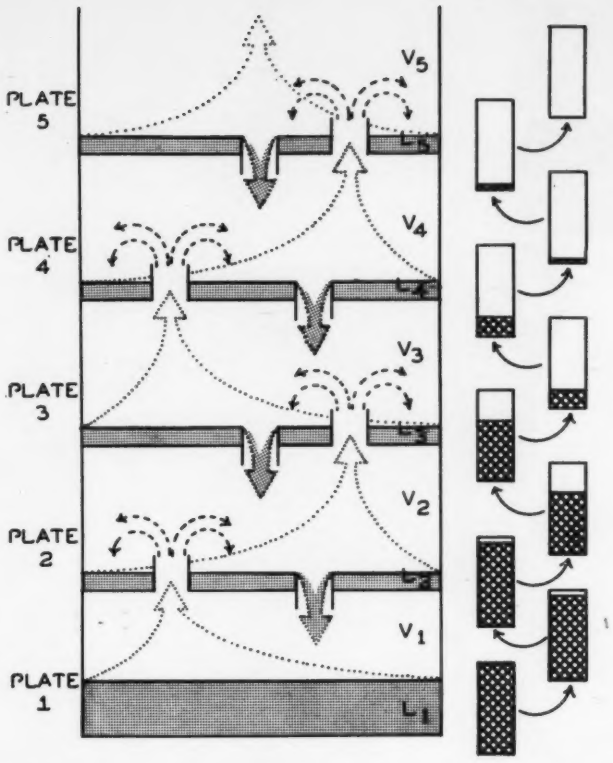
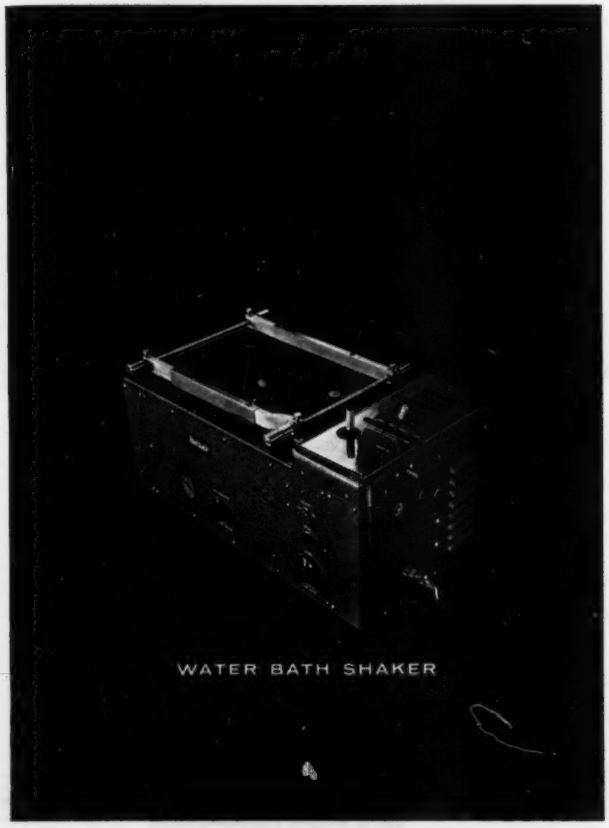


Figure 5. Illustrating the principle of counter-current fractionation as applied in a distilling column. Vapor travels upward while a counter-current of condensate travels in the opposite direction. At each plate, equilibration is established between liquid and vapor.



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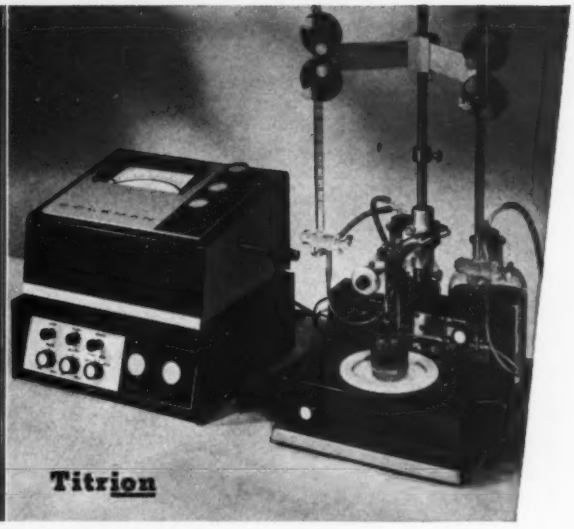

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diagrammatic form that as the first portion of solution contacts the adsorbent, all four species are adsorbed. As additional solution reaches the sites where adsorption has already taken place, the A molecules will first displace the most loosely-held D's, then in turn C's and B's. The displaced molecules move on and become adsorbed on vacant sites, where they remain until displaced again. As the sample continues to enter the adsorbent, the components continue to adsorb, and displace, or be displaced, with the result that a molecular sorting-out occurs, the least strongly-held species traveling at the forefront of the solute zone, followed in order of adsorbability by the other species.

The composition of the effluent emerging from the adsorbent bed varies during the course of the frontal development in the manner shown in Figure 8. At first, when there are vacant sites in the adsorbent, pure solvent issues. This is followed by the emergence of the most weakly adsorbed constituent, unaccompanied by any of the other species. At this point, there are no vacant sites left in the adsorbent. As more sample is passed into the adsorbent, strongly-adsorbed species displace less strongly-held molecules, and when there is no more of the least adsorbable species (viz., D) left on the adsorbent, the effluent begins to contain some of the next most adsorbable component (viz., C). Eventually, all adsorption sites will be occupied by the most strongly held species (viz., A), and from then on the sample will pass through the adsorbent without any change.

Types of Systems

Chromatographic techniques have been devised for practically every type of two-phase system; probably the only exception is the utilization of distribution between two solid phases, since the slowness of diffusion in solids makes the time for the attainment of partition equilibrium impractically long. In several cases, specific names are applied to the chromatographic technique to designate the type of two-phase system employed.

There are two common forms of liquid-liquid chromatography. If both liquids are mobile phases, the technique is known as *counter-current extraction*. If one of the liquid phases is present in relatively small amount, and is supported on the surface of an inert solid carrier, the technique is generally designated as *partition chromatography*. In partition chromatography, only a liquid and a solid phase are visibly evident, but the distribution equilibration actually occurs between two liquid phases (the free and supported liquids, respectively).

When selective distribution of components at a liquid-solid interface is utilized for chromatographic purposes, the technique is called *adsorption chromatography*. This technique looks superficially the same as partition chromatography, but differs in that the solid phase is not serving as an inert carrier for a liquid film, but instead is active in the distribution process.

(*Continued on page A524*)

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If the two-phase system involves the equilibration between a gas and a solid, the technique is called *gas-adsorption chromatography*, or GAC. Distribution techniques based on equilibration between a gas and a liquid which is supported on an inert solid are properly referred to as *gas-liquid partition chromatography*, or GLPC. The less precise terms vapor-phase chromatography (VPC), and gas chromatography are sometimes used when GLPC is meant, but since these terms could also apply to gas-adsorption chromatography, they should be avoided unless the context makes clear the type of two-phase system under consideration.

Production of Counter-Current Flow

The type of force that may be used to produce the counter-current movement of one phase relative to the other depends upon the state of aggregation of the phases and upon their physical properties. If one phase is a gas or vapor, compression or heating conveniently produces a pressure excess over atmospheric pressure that provides the driving force to move the gas over the solid, or solid-supported liquid phase.

In liquid-liquid partition, when both liquids are mobile rather elaborate mechanical contrivances are necessary to shake the liquids together, separate them, and move the separated phases relative to each other. If one of the liquids is supported on an inert solid so that it is immobilized, the second liquid can be allowed simply to flow down over the first

(*Continued on page A526*)

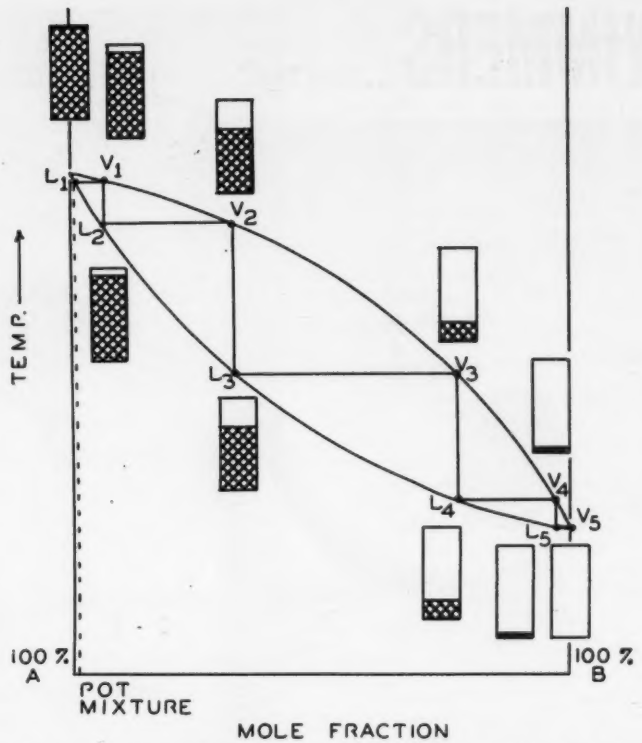

Figure 6. Illustrating the source of the progressive enrichment of the vapor phase in the course of the counter-current fractionation depicted in Figure 5. Liquid of composition corresponding to L_1 is in equilibrium with vapor of composition V_1. When this vapor condenses, it forms liquid L_2, which establishes its equilibrium with vapor V_2, etc. Although the pot mixture contains mostly component, A, vapor V_5 is practically pure component B.

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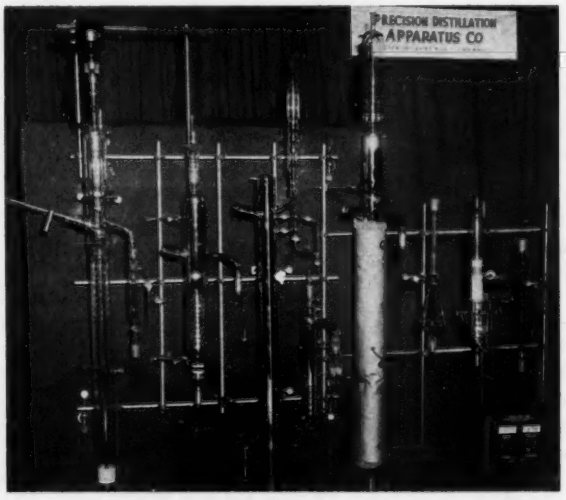

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under the influence of gravity. The force of gravity can be aided, and the chromatographic distribution process speeded up, by employing a gas under pressure as a piston, or the centrifugal force generated by rapid rotation, to push the mobile liquid past the solid. The same considerations apply to adsorption chromatographic techniques.

The solid phase, or the solid support for a liquid phase, may be a fibrous, absorptive material, such as paper strips. In this case, the capillary action due to the surface tension of the liquid phase can be used very effectively to cause the liquid to pull itself through the pores formed by the interstices between the fibers, thereby producing the counter-current motion.

If two phases in contact with each other do not move in a relative sense, but the sample components in one of the phases are caused to move in a given direction by a force that acts on them but not on the bulk phases, the net result is in every sense equivalent to a relative motion of the two phases. For example, the action of an electric field on charged species in solution can produce an electromigration of these species which will move them along an interface across which partition is occurring. This will produce the same kind of concentration distribution as is illustrated in Figure 4. Chromatographic techniques in which the counter-current motion is produced by, or aided by, an electric field are called *electrochromatography*. The term *electrophoresis* applies to the migration of charged species in a single phase under the influence of an applied electric field. Thus, electrophoresis in a homogeneous medium, or zone electrophoresis in which discrete zones of solute are caused to migrate and fractionations of components due to differences in electromobility are achieved, is not a chromatographic technique. However, *paper electrophoresis* as it is commonly carried out does involve partition or adsorption between the electrolyte solution and the solid substrate, and is a form of electrochromatography. A technique of zone electrophoresis in which the liquid medium is immobilized and supported by a solid such as paper, gelatin, starch gel, etc., is sometimes called *ionography*.

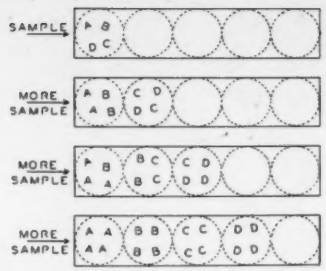

Figure 7. Diagrammatic representation of the mechanism of a frontal development. The sample is assumed to contain equal concentrations of molecules *A*, *B*, *C*, and *D*. The strength of adsorption decreases in the order: $A > B > C > D$. As more sample is added, *A*'s displace all other molecules, pushing them ahead; *B*'s displace *C*'s and *D*'s; *C*'s displace only *D*'s.

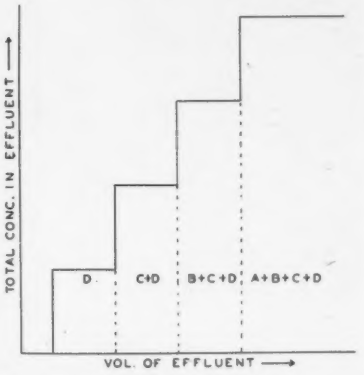

Figure 8. In a frontal analysis, the most weakly adsorbed component breaks through first, followed in incremental succession by the progressively more strongly adsorbed components. When the adsorbent is saturated with the most strongly adsorbed molecules, the sample comes through unchanged, and continues to do so thenceforth.

Scale of Chromatographic Separations

Counter-current distribution techniques are principally suited for the resolution and analysis of small samples because of the requirement that the sample must be so limited in amount relative to the bulk of the two phases between which its components are distributed that characteristic equilibrations for each component can be achieved many times during the relative displacement of the two phases. Thus, these techniques are invaluable for ultramicro and micro scale samples, and successful resolutions of mixtures of a score or more ingredients have been performed

(Continued on page A531)

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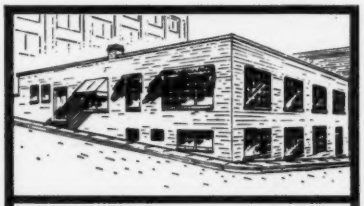

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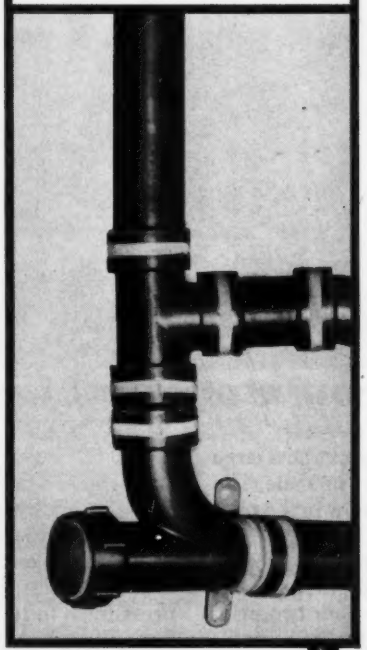

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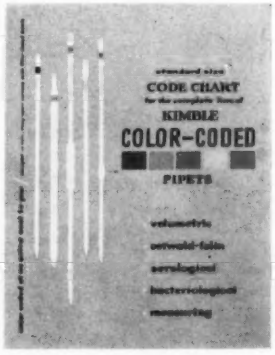

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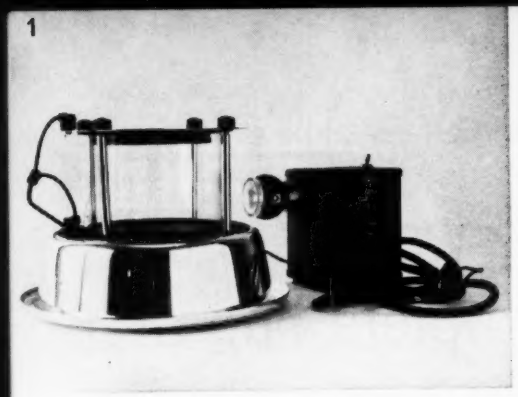

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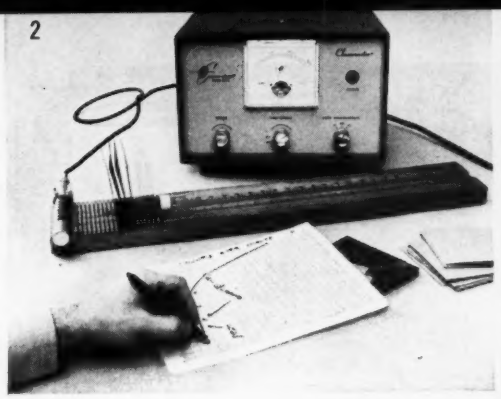

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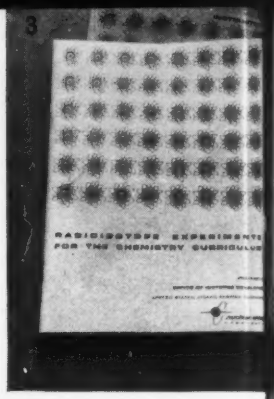

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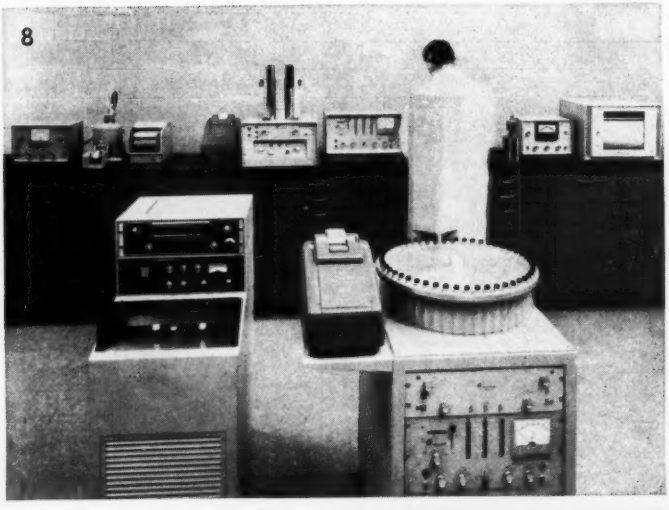

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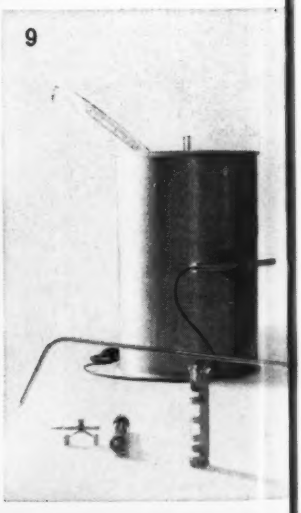

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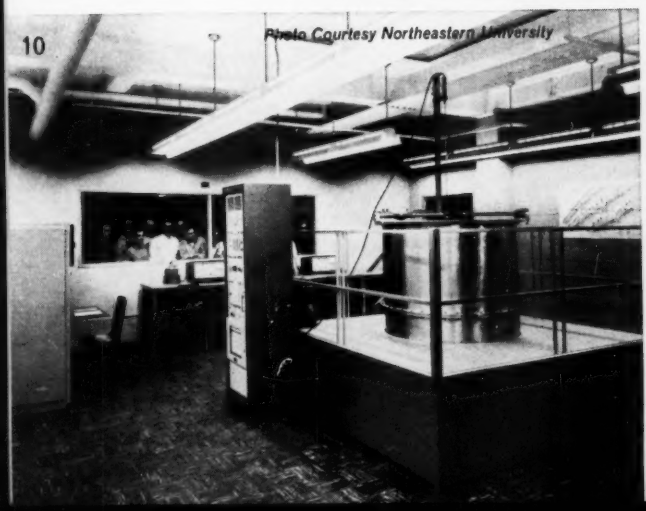

Photo Courtesy Northeastern University

11

Photo Courtesy Shell Oil Company

How to bring radioisotopes into your chemistry curriculum

Read how these Nuclear-Chicago products help to enlarge the scope of chemical education and also serve graduate and faculty research needs

RADIOISOTOPE procedures offer chemistry students penetrating insights into the laws of nature. Forward-looking educators are using the equipment illustrated at the left in lectures, for student experiments, and in research. These eleven photographs suggest the scope and completeness of Nuclear-Chicago's instrument line for teaching and research. Systems range from low-cost equipment for demonstration to sophisticated instrumentation for fundamental investigation. All are characterized by reliability, precision, versatility, and inherent safety.

Chemistry departments now employing radioisotope techniques will want to consider program expansion to take advantage of the newer products illustrated. Others may well regard the apparatus presented here as a planning guide.

Low-cost demonstration equipment for the nuclear age became a reality with the introduction of Nuclear-Chicago's Cloudmaster® and Classmaster®. The Cloudmaster (figure 1) is a continuously sensitive cloud chamber that provides a spectacular display of ionization tracks caused by alpha, beta, gamma, and meson radiation. The Classmaster (figure 2) is a complete system for demonstrating radiation absorption, scattering, geometry effects, coincidence loss, etc. In addition, it permits simple tracer experiments using license-exempt radioisotopes. Both instruments enjoy wide acceptance because of their simplicity of operation and the vivid, easily-understood presentations they make possible.

Thirty-three student experiments that fit naturally into the chemistry curriculum without requiring changes in basic course content are contained in a new 187-page experiment manual and 60-page companion volume of instructor notes (figure 3) developed by Nuclear-Chicago under contract with the Atomic Energy Commission. Model 4001 nuclear training system (figure 4) is offered with isotopes, sample preparation equipment, and instruments to perform all the experiments covered in the manual. The system features ease of sample preparation using the chimney funnel shown in figure 5.

Model 4001 is supplied with the long-lived radionuclide set RNS-110 (figure 6) and the radionuclide certificate book RCB-1 (figure 7) with which the instructor can order short- or long-lived isotopes as they are needed. Neither requires an AEC license to purchase or use, and both are available individually.

As student mastery of radioisotope procedures advances, a chemistry department will next want to consider establishing a complete nuclear laboratory for precision handling, detection, and counting of beta- and gamma-emitting isotopes. Such a facility has valuable application to qualitative and quantitative studies at both the graduate and undergraduate levels. A typical installation is pictured in figure 8. In the left foreground is the Model 701 liquid scintillation detector with Logic scaler, and at the right is the Model C120 automatic sample changer for gamma-emitting liquids or solids in test tubes. In the background. from the left, are the Actigraph II® system for scanning radiochromatograms, a low-background automatic sample changer for solid beta samples, and the Dynacon® electrometer for high-efficiency measurement of beta samples in the gaseous phase.

At this stage, the ideally equipped university will lack only the means to create its own radioactive isotopes and to study reactor techniques and fundamental nuclear properties. For these basic needs, Nuclear-Chicago offers a three-step program.

First, undergraduate departments will choose the NH3 neutron howitzer (figure 9) for experiments with low-level neutron sources, production of short-lived, low-intensity radioisotopic tracers, and low-flux activation analysis.

Second, the chemistry, physics, and engineering faculties will often cooperate in establishing an advanced training facility centered on the inherently safe Model 9000 subcritical assembly (figure 10). Characteristics of this water-moderated, natural uranium assembly are nearly identical in nature to those of a full-scale power or research reactor, and the student readily masters the fundamentals of reactor theory and design.

Finally, the long-felt need of university research scientists for a versatile, relatively inexpensive, high-intensity neutron source can be well satisfied by the TNC neutron generator designed and manufactured by Nuclear-Chicago's subsidiary, Texas Nuclear Corporation (figure 11). Providing a controllable yield of 4×10^{10} neutrons per second and a thermal neutron flux of greater than 10^8 using an appropriate moderator, the generator is widely employed in activation analysis, process control, applied research and development, and fundamental investigation.

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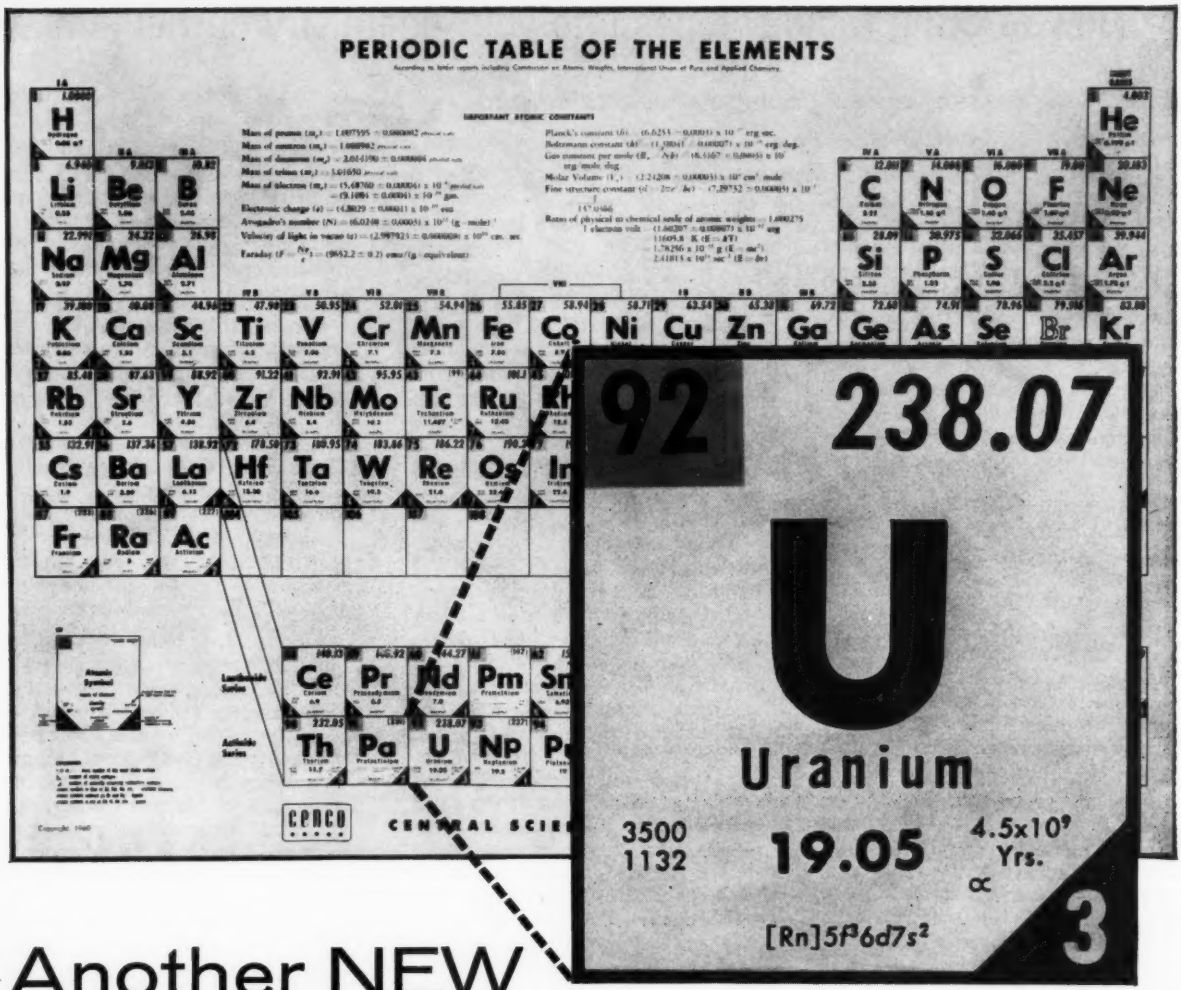

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on micrograms of material. The lower limit of sample size is determined principally by the sensitivity (or signal-to-noise ratio) of the detection method used to determine the concentration distribution of material after the chromatographic development has taken place. The upper limit is determined by the dimensions of the two-phase system, the difference in distribution coefficients of the components of the sample, and the sharpness of separation required.

The largest samples that are usually handled in laboratory-scale chromatographic equipment are of the order of tenths of a gram. For larger samples, the equipment is termed a *preparative chromatograph*, and the capacity for handling large samples is achieved either by scaling up the dimensions of the entire apparatus, or by employing a scheme for continuously introducing the sample in small increments, while removing the distributed fractions as they are produced.

Components of Chromatographs

The essential instrumentation required for chromatography includes: (*a*) the two phase system; (*b*) a container for the two-phases so designed that equilibrations can occur during relative displacement of the phases; (*c*) means of introducing

(*Continued on page A532*)

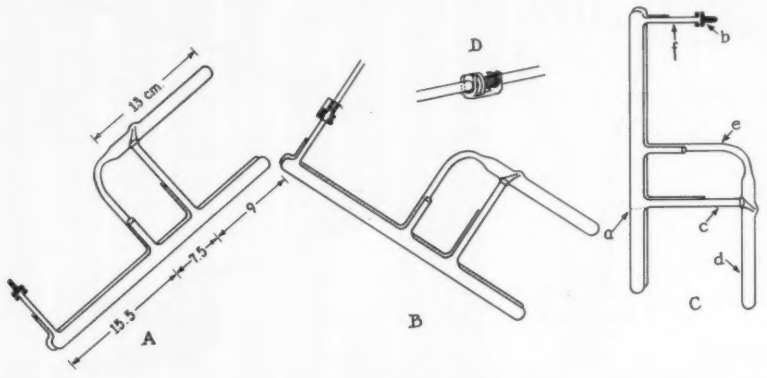

Figure 9. Design of component extraction vessels for Craig counter-current extraction apparatus. Positions *A* and *B* are for mixing of the two liquid phases; position C followed by *A* is for transfer of phases to and from adjoining units.

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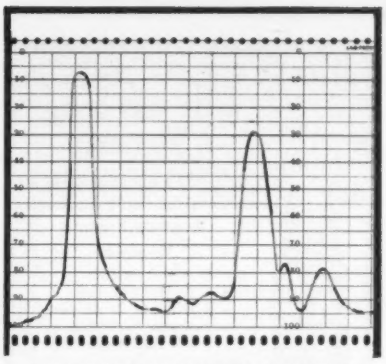
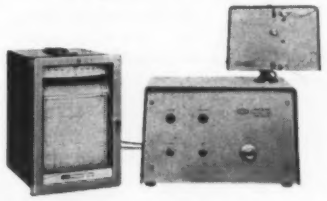


THIS UVICORD DIAGRAM SHOWS: Peptides released from α-casein through enzymatic degradation by action of Lactobacillus lactis. Six peaks are separated by molecular sieving through a column of SEPHADEX G50.

Column: Sephadex G50, 2 × 110 cm
Sample: 180 mg mixture of peptides with varying molecular size
Buffer: glycine-0.1-N sodium hydroxide, pH 9.9
Flow rate: 30 ml/h at 0.3 atm. air pressure on buffer
Chart speed: 10 mm/h

* **A FASCINATING SEPARATION RESOLVED WITH THE LKB 4701 UVICORD**



Connected directly to the column outlet, the *Uvicord* measures continuously and without appreciable distortion the UV absorption of an eluate, revealing the interesting components even when present in small quantities.

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0.036 % thyrosine in 0.1-N NaOH, 1 mm cell ≃ 100 mm pen deflection (E = 1)
Example 2. 0.02 % serum albumin in 1-% NaCl, 5 mm cell ≃ 10 mm pen deflection (E = 0.05).

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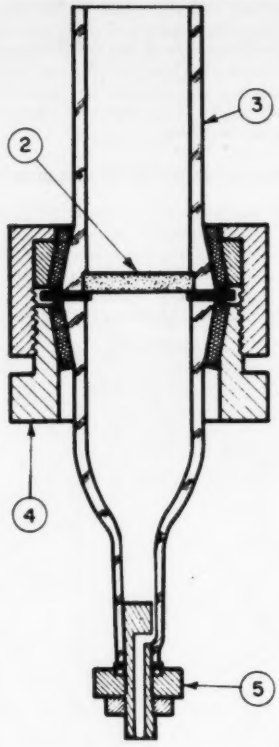

Figure 10. RSCO chromatography column. 2. sintered glass disc for support of adsorbent bed. 3. column with flanged ends. 4, threaded coupling. 5. flow control valve with Teflon barrel.

the sample; (*d*) means of adjusting and controlling the force field which produces the counter-current motion of the two phases; (*e*) means of detecting the distributed material after completion of the chromatographic development. In addition, it is often found necessary to employ specialized instrumentation for (*f*) preparing the sample so that it is suitable for chromatographing (e.g., desalting of amino acid or protein samples); (*g*) modifying or changing the properties of one of the phases continuously during the development process (e.g., increasing the polarity of the liquid phase at a programmed rate); and (*h*) automating the collection and read-out of the analytical data.

The Two-Phase System

The choice and chemical preparation of the two-phase system for the resolution of a given sample is a sophisticated chemical problem, for it involves consideration of the interactions between each phase and the various sample components, as well as the interaction between the phases themselves, and the effect of this on the distribution equilibria that will be established. A number of comprehensive summaries of the results and generalizations arrived at by chromatographers are available, and should be consulted before attempting to chromatograph a new type of sample. For counter-current extraction procedures, see Craig, L. C. and Craig, D., "Laboratory Extraction and Counter-

(*Continued on page A534*)

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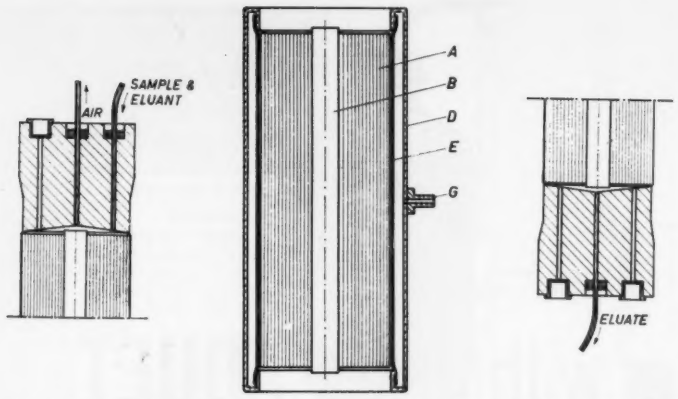

Figure 11. Schematic diagram of the LKB ChroMax pressurized paper roll chromatography column. *A.* paper roll. *B.* polyethylene rod. *D.* steel cylinder. *E.* plastic bag. *G.* air inlet valve.

current Distribution," in Weissberger, A., editor, "Technique of Organic Chemistry," Interscience Pub., N. Y., 2nd Ed., 1956, vol. III, part I, Chap. II. For adsorption and partition procedures, see Cassidy, H. G., "Adsorption and Chromatography," in *ibid.*, vol. V. For a compilation of paper chromatographic procedures, see Balston, J. N. and Talbot, B. E., "A Guide to Filter Paper and Cellulose Powder Chromatography," H. Reeve Angel and Co., Ltd., London, 1952. Other references will be found in the Bibliography section.

Containers

For liquid-liquid counter-current extraction, the two liquid phases are contained in a separatory funnel which can be shaken to produce equilibration, and from which the two liquid phases can be removed and mixed with different portions of liquid. The type of container designed by L. C. Craig is shown in Figure 9. This vessel operates by being rocked several times from position *A* to *B* and back again, to produce equilibration. Upon standing in position *B*, the two liquid layers separate; the vessel is then tipped to position *C*, whereupon the upper layer passes through the decantation tube *c* into compartment *d*. Upon tipping the vessel back to position *A*, the liquid in *d* drains through the transfer tube *e* into the next extraction vessel of the series, where it mixes with a portion of the second liquid phase. With this design, a large number of vessels can be connected together in a compact, convenient unit.

If one of the phases in an adsorption or partition procedure is a granular solid, a simple column packing is adequate support for the fixed phase. A number of convenient column designs especially suited to chromatography with a flowing liquid phase are commercially available. Figure 10 shows the chromatographic column of Research Specialties Co. (RSCO), Richmond, California. The parts are interchangeable and replaceable, and the flow control valve, bed support disc, and column proper are held together by a hand-tightened, threaded aluminum coupling (typical price of a complete assembly for an 18-inch long, 1-inch diameter column: $15).

Several rather specialized types of column containers are also produced. A tightly-wound roll of filter paper is employed as the column packing in the ChroMax pressurized column made by LKB-Produkter, Sweden and distributed in the U. S. by LKB Instruments, Inc., Washington 14, D. C. (price: about $300). The construction of this column is shown in Figures 11 and 12. The paper roll is inserted in a polyethylene or rubber bag, and this is placed inside a steel cylinder. Gas pressure is built up in the annular space between the inside of the steel cylinder, and the outer surface of the plastic bag. This forces the plastic wall against the paper roll, and serves to press

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Figure 12. Photograph of LKB ChroMax column and components

out and eliminate any non-homogeneities in the paper roll which may tend to develop due to irregular swelling caused by the absorption of solvents. This column permits chromatographic procedures worked out with single paper sheets to be scaled up without modification to the preparative range (up to gram quantities of sample).

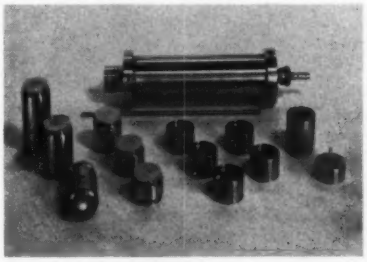

Figure 13. LKB Composite Chromatography Column, consisting of a graded series of sections with diminishing lengths and inner diameters.

Another unique design of a column container for a granular adsorbent packing is the LKB Composite Column ($250), illustrated in Figure 13. It consists of a graded series of sections having successively decreasing lengths and diameters. Each section connects to the next through a capillary coupling; as the moving fluid passes through the capillary, localized mixing occurs and as a consequence any non-uniformities in the advancing front of the solute zones are straightened out, and the separations obtained are sharpened.

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Next: Continuation of survey of chromatographic equipment.

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▶ Fluid process streams which previously could not be measured accurately because they were highly viscous, extremely dark, opaque or because the physical character changed when a sample was removed from the process can now be measured by a new In-Line Refractometer offered by Waters Associates, 45 Franklin St., Framingham, Massachusetts.

The significant feature of the Waters In-Line Refractometer is that it measures the refractive index at the surface of a liquid sample. Since surface reflection requires no penetration of the light beam into the solution the instrument may be used for highly opaque and various murky solutions and suspensions as well as transparent solutions.

▶ International Equipment Co., 1284 Soldiers Field Rd., Boston 35, Massachusetts, offers the International Model HR-1 high speed, high capacity, refrigerated centrifuge. Five angle heads are available, ranging from 12-place 7 ml. to 6-place 250 ml. The standard 8-place 50 ml. head offers 40,180 × G at the maximum speed of 18,500 rpm. A new Continuous Flow System is available for the HR-1 employing an entirely new method of high speed continuous flow centrifugation. Temperatures as low as −20°C are controlled within ±1°C.

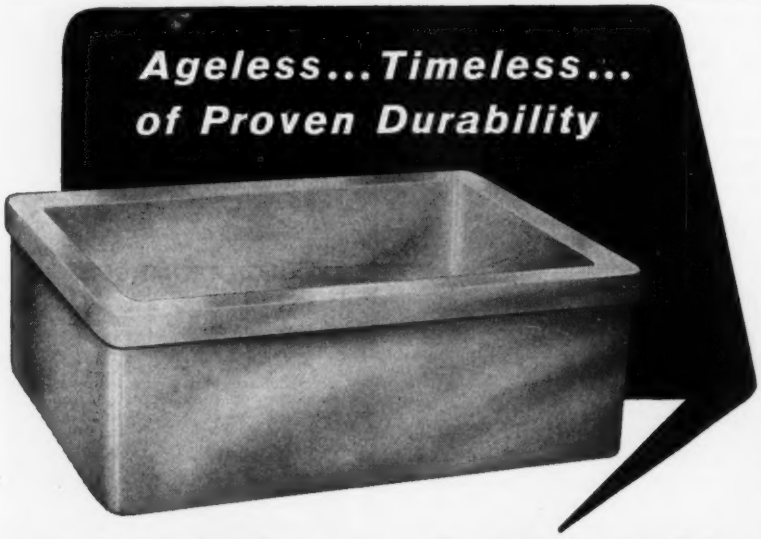
▶ The Nalge Co., Inc., 75 Panorama Creek Dr., Rochester 2, New York, announces a new series of Graduated Cylinders. As part of a continuing program of product improvement, the Company has completely redesigned and retooled their line of graduated cylinders. Utilizing new Nalge techniques, these new polypropylene cylinders can be molded to unusual accuracy. A random sampling by an independent, nationally - known testing laboratory shows the new graduates to be well within National Bureau of Standards Class A specifications for accuracy.

▶ Roger Gilmont Instruments, Inc., 1 Great Neck Rd., Great Neck, New York, announces the development of a new laboratory manometer. The age - old problem of obtaining a positive closed-end has been solved with the novel application of modern "O" rings and precision bore tubing. Other features include a permanent ceramic scale with white background for easy accurate reading.

▶ A new 6-in. laboratory electromagnet is announced by Varian Associates, 611 Hansen Way, Palo Alto, California. Designated the V-4007-2, this new six-inch magnet features pole pieces of which the air gap width (which dictates field strength) may be continuously adjusted from $^1/_{32}$ to $5^1/_2$ in. by means of an integral gap-adjusting mechanism. The V-4007-2 enables scientists to examine samples of widely differing sizes at many desired magnetic field strengths.

▶ The addition of a simple accessory to graduated cylinders now means that laboratory technicians can work with a

(*Continued on page A537*)



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cylinder that will bounce and not break if it is accidentally tipped over.

Kimble Glass Co., a subsidiary of Owens-Illinois, is now packing "Safe-Gard" plastic bumpers at no increase in price with its Kimax graduated cylinders. The yellow plastic ring is cut on one edge to allow it to be slipped easily around the cylinder. Once on the cylinder, it fits snugly and will protect the cylinder from breakage when accidentally tipped over, the plastic cylinder absorbing the impact instead of the rim or pourout.

▶ Worden Laboratory, 695 Rocky River, Houston 27, Texas, offers an "Engineering Kit" to demonstrate the scope of fused quartz springs and fibers. It consists of an assortment of fused quartz products for experiment necessitating drift free measurements of small forces.

New Literature

● Write Black Light Eastern Corp., 4 Manhasset Ave., Port Washington, Long Island, New York, for copies of *Chromato-Vue* which describes the technical and service data pertinent to their portable ultra violet fluorescence analysis cabinet.

● A new pH Recording Adapter for use with high quality potentiometric recorders has been developed by E. H. Sargent and Co., 4647 W. Foster Ave., Chicago 20, Illinois. Ask for *Bulletin RA*.

● National Appliance Co., 7634 S.W. Capitol Highway, Portland 19, Oregon, offers a 22-page bulletin on techniques for experimentation on bacteria, tissue, and virus culture in oxygen free atmosphere. Eight pieces of apparatus designed for carbon dioxide incubation are also described.

● More than 20,000 carefully evaluated items are listed in the 61st anniversary edition of their catalogue, *Scientific Apparatus and Reagents*, now being distributed by the Arthur H. Thomas Co., Philadelphia, to scientists throughout the free world. Format of the encyclopedic, 1148-page volume has been revised to render it lighter in weight for easier handling, but extensive listings of the latest laboratory instruments with full complement of accessories have been retained. Special attention was given to the Index which is replete with cross-references.

● Cole-Parmer Instrument and Equipment Co., 7330 N. Clark St., Chicago, 26, Illinois, has just released its new *1961–62 Catalogue*, a virtual reference book for the chemist, engineer, biologist, and related user of scientific equipment. It contains a most comprehensive compilation of up-to-date instruments and equipment for general research, clinical study, production, quality control, and pilot plant operation. Copies are available on request.

● Mallinckrodt's 1961 *A.R. Laboratory Chemical Catalogue* features a 20-page

(*Continued on page A538*)

The Improved Welch
CATHETOMETER

Significant recent engineering improvements in this instrument insure better performance than ever and more years of satisfactory service. This cathetometer is designed for precise measurement of vertical distances through a range of 100 cm. It is particularly useful where the object to be observed is not conveniently accessible, or may easily be disturbed by physical contact, or must be located in a position too hazardous to permit the observer to be in close proximity.

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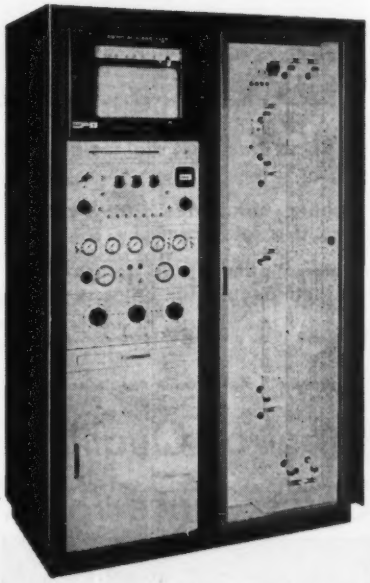

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technical information section and convenient alphabetizing of pages for quick, easy reference. Copies may be obtained from Mallinckrodt Chemical Works, 2nd and Mallinckrodt Sts., St. Louis 7, Missouri, and 72 Gold St., New York 8, N. Y.

● Technicon Controls, Inc., Research Park, Chauncey, New York, offers a two-page data sheet outlining a continuous automatic method for determining total acidity.

● Available immediately is a new, 24-page, catalogue *P61* of laboratory plasticware from Will Corp., Box 1050, Rochester 3, New York. The new brochure features plasticware of all types: beakers, bottles, buckets, funnels, gloves, graduated cylinders, stirring apparatus, tubing, and connectors—even plastic mouse houses.

● Bulletin 10-1, *High Vacuum Valves, Baffles and Traps* is now available from Consolidated Vacuum Corp., 1775 Mt. Road Blvd., Rochester 3, New York.

● General Biochemicals, 680 Laboratory Park, Chagrin Falls, Ohio, offers a 1961 price list of *Special Products for Research.*

● A new catalogue *No. 442* on automatic water service systems, designed with foot control valves and "stream-mated" service fittings, is now being issued by T & S Brass and Bronze Works, Inc., 128 Magnolia Ave., Westbury, Long Island, New York.

● Publication of a new catalogue on high vacuum technology and products has been announced by the Vacuum Products Division, Varian Associates, 611 Hansen Way, Palo Alto, Calif.

● For copies of the *Glossary of National Organic Chemicals* write Department NA60, National Aniline Division, 40 Rector St., New York 6, N. Y.

● Catalogue No. 161 *Plastics and Other Materials for Science and Industry,* is available on request from Bel-Art Products, Pequannock, New Jersey.

● Applied Science Laboratories, Inc., State College, Pennsylvania, announce a variety of new gas chromatography supplies in the spring issue of their *Gas-Chrom Newsletter.*

● *The Corrosion Resistance of Zirconium* is a new publication offered by the Zirconium Association, 2130 Keith Building, Cleveland 15, Ohio. This 14-page book contains detailed graphs for over 100 highly corrosive media in concentrations up to 100 per cent and temperatures up to 400°F. The data contained in the book was compiled from information developed by the countries leading industrial corrosion testing laboratories.

(*Continued on page A541*)

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** The Picker Nuclear Division is prepared to help interested institutions in drawing up training programs in the use of nuclear techniques in biology, chemistry, medicine, agriculture, physics, and other fields.*

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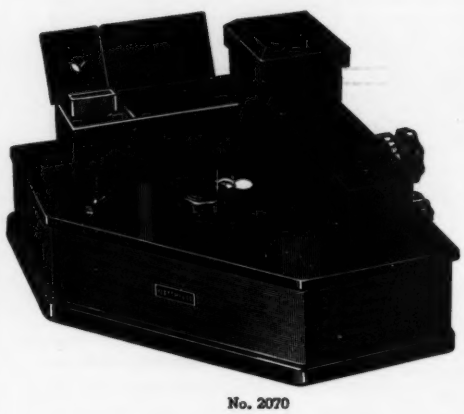

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Out of the EDITOR'S BASKET

● Gow-Mac Instrument Co., 100 Kings Rd., Madison, New Jersey, offers a new literature release entitled *Gas Density Detectors*. The brochure describes a gas density balance with no moving parts.

● A comprehensive, new two-color chart that provides a comparison of the physical properties of over 200 commercially available common organic solvents is now available from the Solvents and Chemicals Group, 2540 W. Flournoy St., Chicago 12, Illinois.

● The recently released Burrell *Catalogue 92* offers and describes a complete line of heating equipment and apparatus for the laboratory. Copies are available from Burrell Corp., 2223 Fifth Ave., Pittsburgh 19, Pa.

● The following publication may be ordered from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. *Standard X-ray Diffraction Powder Patterns*, by Howard E. Swanson, Marlene I. Cook, Eloise H. Evans, and Johan H. deGroot, National Bureau of Standards Circular 539, Volume 10, issued September 23, 1960, 61 pages, 0.40 cents.

● Kewaunee Mfg. Co., Adrian, Michigan, and its affiliate, Kewaunee Technical Furniture Co., Statesville, North Carolina, announce the publication of a new 92 page catalogue, *Section 5-Wood Educational Laboratory Furniture*, 1961 edition, for secondary schools and junior colleges. Featuring many typical illustrations and the complete Kewaunee line of functionally - designed, craftsman - built educational science furniture, the new catalogue is a helpful planning aid.

Illustrated and described in detail are instructor's desks, perimeter tables, combination science tables, cabinets and casework, laboratory sinks, fume hoods, special purpose units, service fixtures, and accessories. Dimension drawings and specifications are included to aid in efficient planning of science laboratories. For additional information, write to Kewaunee Technical Furniture Co., 3003 W. Front St., Statesville, North Carolina.

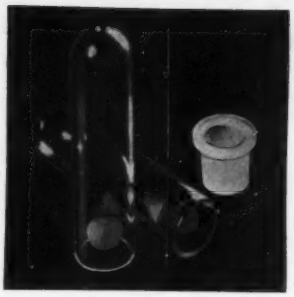
● A new 32-page Bantam-ware catalogue is now available from Kontes Glass Co., Vineland, New Jersey. Designated *Catalog BW-2*, the publication has over 150 new items in the Company's expanding line of small organic glass apparatus. Three new kits are featured.

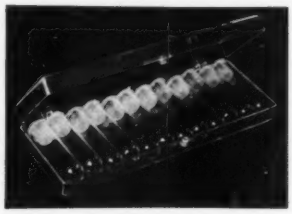
Prominent in the listing of new products are items for distillation, extraction and concentration. Volumetric glassware and micro syringes, needles and adapters are also listed for introducing and withdrawing samples.

The catalogue contains complete descriptive information and current prices.

● Expanded data on corrosion resistance

(*Continued on page A542*)





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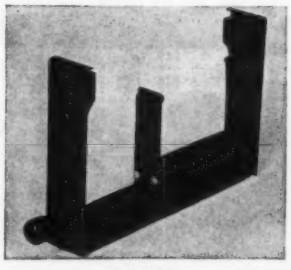

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Out of the EDITOR'S BASKET

and thermal expansion of 32 commercial glasses are major additions to a revised booklet published by Corning Glass Works.

The brochure entitled, *Properties of Selected Commercial Glasses* contains more complete information on evaluating the hardness of glass, a new numerical code system which shows the resistance of each glass to weather, water, and acid and standard sections on mechanical, electrical, and optical properties; thermal stress; heat transmission and viscosity of the glasses.

The revised bulletin, coded *B-83*, is available upon request on company letterhead from Corning Glass Works, Corning, New York.

● The *1961 Sigma Catalog* is now available. It contains information on aerosols, amino acids and derivatives, buffers, carbohydrates, carcinogenics, chromogenic substrates, enzymes, enzyme assay procedures, fluorinated compounds, histochemicals, ion exchanger resins, nucleotides and bases, steroids and free technical bulletins. Reagents and special kits for the Sigma Enzyme Assays are also listed.

All items are listed alphabetically on white pages. Separate lists of "classified" items are on yellow pages.

For further information, write Sigma Chemical Co., 3500 DeKalb St., St. Louis 18, Missouri.

● Typical chemical reactions of hexachlorocyclopentadiene are featured in a new technical bulletin published by Hooker Chemical Corp.

C-56 is the registered trademark for this highly reactive compound. Among end products derived from C-56 are pesticides, non-flammable resins, and chemical intermediates having potential application as fungicides, germicides, pharmaceuticals, dyes, and resins for paints.

In addition to the typical reaction products illustrated by their structural formulas, the publication covers physical data on C-56 toxicity, and recommendations for handling the product.

Copies of the publication, *Bulletin 65*, may be obtained by writing to Hooker Chemical Corp., Niagara Falls, New York.

New Compounds and Chemicals

◇ City Chemical Corp., 132 W. 22nd St., New York 11, N. Y., has experimental quantities of cadmium oxalate, cupric glycolate, sodium mandelate, and stanuous pyrophosphate now in stock.

◇ "Baymal" colloidal alumina, a versatile addition to the inventory of basic chemicals available to industry was announced recently by DuPont's Industrial and Biochemicals Department. Research chemists who developed "Baymal" say it has more unique properties and use possibilities than any chemical to emerge from their laboratories in many a year. To illustrate this versatility they point to the fact that in one form "Baymal" is hard

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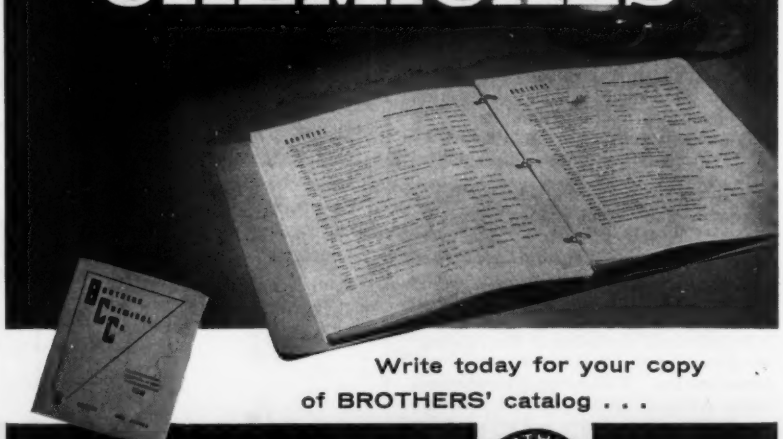

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enough to machine steel or cut glass, while in another form it can be used to improve the smoothness of a skin lotion. Chemically, "Baymal" is a white, free-flowing powder, consisting of clusters of minute fibrils of boehmite (AlOOH) alumina.

Miscellany

★ *Science as a Way of Life* by Ellsworth S. Obourn is an interesting consideration of the variety of problems inherent in improving science education in elementary and secondary schools. It includes a check list of questions that might help you evaluate the science programs in your own school. The 25-page pamphlet is published by the U. S. Department of Health, Education, and Welfare and is available from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.

★ If you are interested in getting a research contract from any of the governmental agencies, the newly revised third edition of *Federal Agencies Financing Research* should prove highly useful. Priced at $1, this 26-page report on agencies, scientific fields, and suitable forms of contract application is available from the Social Legislation Information Service, Inc., DuPont Circle Building, Washington 6, D. C. SLIS is a non-profit organization that reports on Washington developments without taking any position for or against particular legislation.

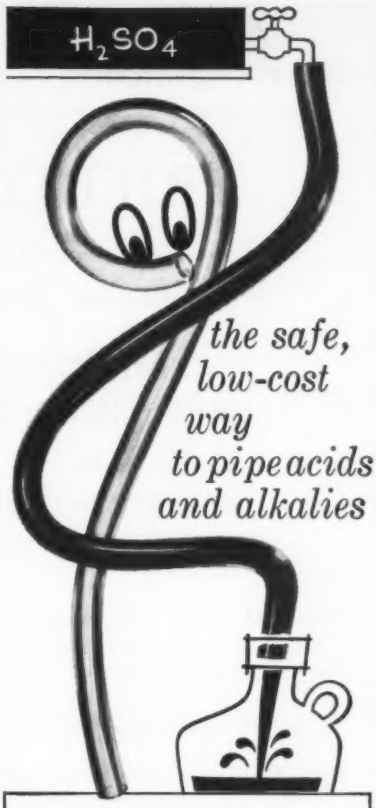
★ A new series of seven educational films to help introduce high school physics students to studies of radiation has just been produced by Cenco Educational Films, 1700 Irving Park Rd., Chicago 13, Illinois.

The series, titled "Nuclear Radiation," explains to the student the different types of radiation (light, radio, X-ray, nuclear, etc.), explains how each is detected, and how each is used for different purposes—in medicine, industry, studies of the earth, studies of outer space. Fact and fiction of nuclear fallout are also discussed.

★ Mr. Howard J. Francis, Jr., Vice Chairman, Intern. Symp. on Microchem. Techniques, c/o Pennsalt Chemicals Corp., P.O. Box 4388, Philadelphia 18, Pennsylvania, U.S.A., announces the International Symposium on Microchemical Techniques to be held August 13–18, 1961, at Pennsylvania State University.

Training manuals and suggested experiments in radioactivity applications are available from: Nuclear-Chicago Corp., 333 East Howard Ave., Des Plaines, Illinois. Picker X-Ray Corp., Nuclear Division, 25 South Broadway, White Plains, New York.

The following government publications are of general interest to users of nuclear radiation instrumentation: National Academy of Sciences—National Research Council, Publication 825, "Source Material for Radiochemistry," Washington, D. C., 1960. U. S. Atomic Energy Commission, "Special Sources of Information on Isotopes," TID-4563 (2nd Rev), Jan. 1, 1960.



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Efficient—Dries all organic liquids instantly in liquid or vapor phase. Gases retain only 0.005 mg. H_2O per liter.

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Regenerative—Repeatedly after any normal use, by dehydration at 200 to 225°C.

Economical—Lowest priced high grade desiccant. Available in quantity for Industrial Processes.

References

(1) Ind. Eng. Chem. 26–653 (June, 1933)

(2) Ind. Eng. Chem. 25–1112 (Oct., 1933)

(3) National Bureau of Standards Journal of Research 12-241 (Feb. 1934, R.P. No. 649)

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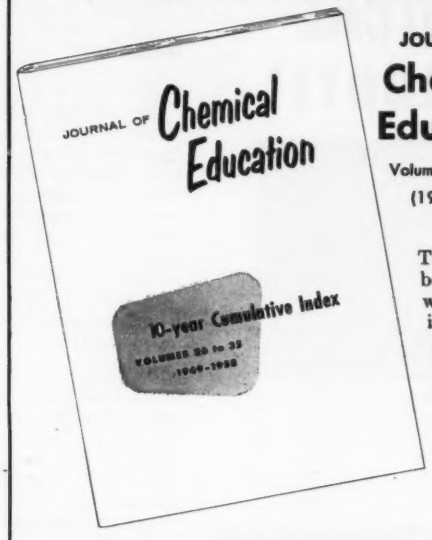

JOURNAL OF Chemical Education

Volumes 26 through 35
(1949–1958)

This index has been prepared with the special interests of the J.-Chem.-Ed. user in mind. Not only are the conventional entries made by author, title, and inverted title, but many suggestive subject listings have been used. For example, reference to an article entitled "Infrared Spectra of Chemisorbed Molecules" can be found under "acetylene," one of the compounds discussed. Preparation of the index involved hundreds of hours of careful checking not only of annual indexes, but the original articles for subject content.

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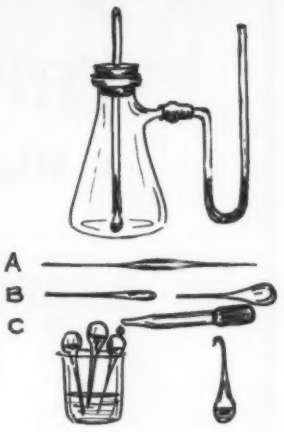
DALTON'S LAW OF PARTIAL PRESSURES

A Chem Ed TESTED DEMONSTRATION

Submitted by: F. B. Dutton, Michigan State University
Checked by: Julian R. Brandou, Traveling Science Teacher Program



PREPARATION

Obtain a 500-ml or larger filter flask with a one-hole stopper to fit. Insert in the stopper a piece of glass tubing which has been sealed at one end and is such length that it will reach nearly to the bottom of the flask and leave a few inches protruding through the top of the stopper. Provide a number of glass ampoules filled with ether or other volatile liquid. For construction and filling of ampoules see remarks below.

Place the stem of an ether ampoule into the open end of the glass tube and slip the flask over the assembly. Adjust the glass tube so that it forms a reasonably snug fit with the ampoule against the bottom of the flask. Be careful not to break the ampoule prematurely.

Bend a piece of glass tubing to make a U-shaped manometer and attach with rubber tubing to the side-arm of the flask. Fill the manometer to an appropriate level with mercury.

DEMONSTRATION

We have a closed system with the pressure inside the system essentially equal to the atmospheric pressure. Strike the closed end of the glass tube sharply breaking the ampoule. The vapor pressure of the ether is now added to the pressure of the air in the flask; the difference between the new pressure in the flask and the atmospheric pressure can be observed on the manometer.

REMARKS

Make the ether ampoule from soft glass tubing by drawing out a small thick-walled bulb (Fig. A). Seal off one end of the tube, rotate it in the flame until soft, and blow a large thin-walled bulb (Fig. B). A number of ampoules may be filled at one time as follows: place them with the open-ended stem down in a small beaker containing a little ether (Fig. C). Place a few drops of ether over the tops of the bulbs; this will cool them sufficiently to draw some of the liquid up into the bulbs. Quickly remove and invert the tubes; boil off the ether by gentle warming. Now place the ampoules once again in the beaker, stem down, and cool; a sufficient quantity of liquid ether will be drawn up. Seal off the ends of the fine capillaries in the flame and store the ampoules until needed for use.

CAUTION: *Ether is highly inflammable.*

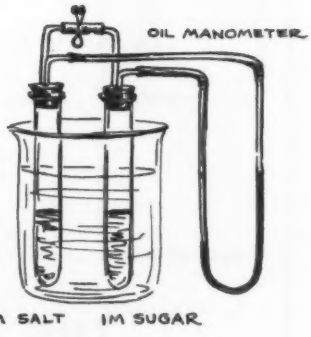
A Qualitative Demonstration of Raoult's Law

A Chem Ed TESTED DEMONSTRATION

Submitted by: Julian R. Brandou, Michigan State University
Checked by: J. F. Janssen, Mason City Junior College, Mason City, Iowa

PREPARATION

Assemble the apparatus as shown, using 7-mm glass tubing, rubber stoppers and connectors, a one-liter beaker, and a pinch clamp. Provide three 8-in. test tubes. Place 50 ml of 1 *M* NaCl solution in one, 50 ml of sugar solution in the second, and 50 ml of water in the third, and label each tube. Place the three tubes into the water bath and bring the bath to a boil for a few minutes just prior to the demonstration.



DEMONSTRATION

Connect the tubes containing water and sugar to each other with the pinch clamp open; next connect this assembly to the oil manometer. As soon as the oil levels are equal, close off the pinch clamp and note the difference as it quickly develops. This difference can be attributed to the difference in vapor pressure of the two solutions in the system. Follow with water and salt solution, and with salt solution and sugar solution.

REMARKS

Ordinary motor oil gives satisfactory results, but the addition of an oil-soluble dye would enhance the visibility of the manometer arms. Raoult's Law is not strictly applicable to concentrated solutions nor to other than ideal solutions; however, with ordinary solvents and non-volatile solutes, qualitative indications of a real difference in vapor pressure may be shown with this apparatus.

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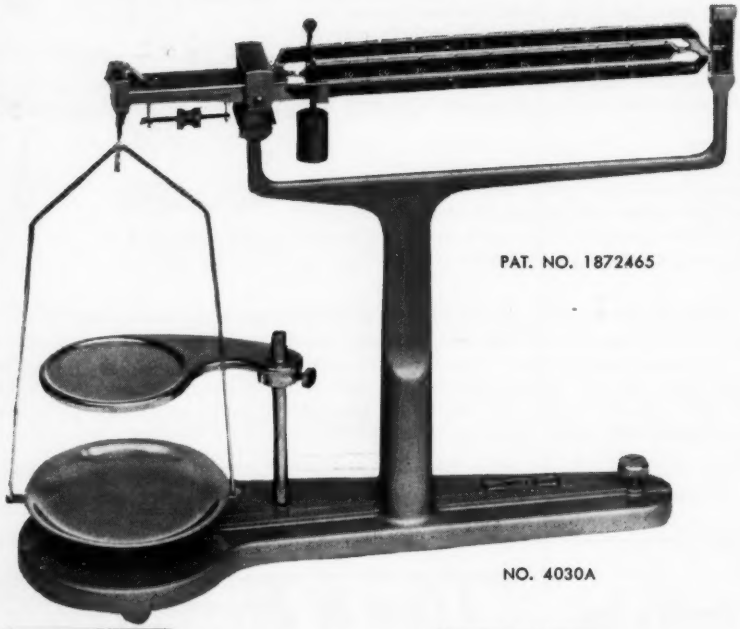

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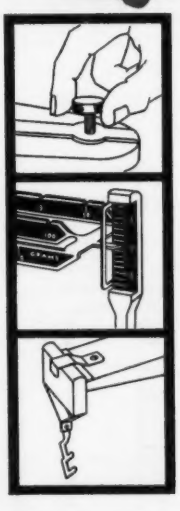

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EDITORIALLY SPEAKING

The National Science Foundation has just released a publication, "Investing in Scientific Progress—1961–1970" (NSF report 61-27), which it hails as a major policy document. The chief premise is that scientific progress concerns us all as individuals and as citizens of the nation. Statistics and interpretations are clearly set forth. The meat of the report, however, is the projection of trends and the analysis of their significance in the decade ahead of us. The reasonableness of these extrapolations is unequivocal; their realization must concern us all.

The report treats two major themes: people and support. The interrelation is crucial. Support, even sufficient to meet projected needs, cannot be translated into people by outmoded and unimaginative science education.

People

U.S. population will increase from 180 to 200 millions from 1960 to 1970. During the same period, world population will increase by 420 million to 3,330 million. *Inference:* Successful maintenance of our role in the world's civilization depends on the quality, not the quantity of our future scientists and engineers.

The percentage of college age young people having bachelor's degrees, now 18%, is doubling every 18 years. The number of science doctorates has increased from 400 in 1920 to the present 6,600 and indicates a doubling every 12 years. While the total labor force, now 74 million, increases at a rate of 1.4% per year, the number of professional scientists and engineers in the labor force, now 1.4 million, increases at a rate of 6% per year. The number of doctoral scientists and engineers, now 87,000, increases at the rate of 7% per year. This will double the present number by 1970. Of all doctoral age young people who score in the top 1% on intelligence tests, less than one in twenty attain science doctorates. *Inference:* The trends shown by statistics suggest that the nation's needs for scientific personnel can be met and "still leave a wide margin of capacity available for intellectual leadership in all professions."

There are about 100,000 on the professional staffs of colleges and universities now teaching science; in 1970 the need will be 175,000. Basic research in these institutions now involves 80,000; in 1970 the need will be 150,000. *Inference:* Numbers alone cannot accomplish the task without developing new methods of fundamental training and greater nurture of creativity during the apprenticeship for research.

Support

The present deficit in laboratory teaching space must be overcome, not allowed to increase. *Inference:* By 1970, $3.5 billion will have to be spent. The present need for research buildings is $500 million. *Inference:* A total of $2.8 billion needs to be provided in the next decade.

Instructional equipment facilities are inadequate at present to the extent of $300 million. Demands for basic research equipment likewise are costly and will increase. *Inference:* Insuring adequate facilities for instruction will cost $200 million a year for ten years; by 1970 we should be investing $180 million a year in equipment for basic research on campuses.

Salaries for the professional staffs teaching science in colleges and universities should increase from the present $800 million to $2,100 million in 1970. Textbook costs and all other operating expenses will increase. The same will be true for the support of basic research. *Inference:* These supporting costs for teaching will rise from the present $1.9 billion total to $5 billion by 1970; for basic research from $760 million to $2,200 million.

The message the report brings to the nation is direct and emphatic:

> Because national welfare, security and prosperity increasingly demand an advancing technology, because technology increasingly needs and springs from science, because scientific talent is a scarce resource, and because we have no monopoly on it, therefore our goal must be to use our abilities to the full.

Professional scientists, especially those whose careers are spent on campuses, can read in this the challenge of two unique obligations. Support can come only from a citizenry convinced that its money is being spent for a vitally necessary program. This conviction can be based only on wide dissemination of knowledge. Clearly, this is a primary role for science education. The second obligation is a more personal concern: superior teaching is essential to superior science. "Time tested" can be no cover-up label for methods that actually are worn out and inefficient. Rapid advances on the research frontier cannot mean a widening gap of ignorance in the classroom.

Investment in complacency cannot produce progress.

R. T. Sanderson
University of Iowa
Iowa City

Principles of Chemical Bonding

An understanding of chemical bonds is so essential to a true appreciation of chemistry that simple yet reasonably valid explanations must continuously be sought and improved. The purpose of this article is to discuss chemical bonding comprehensively, in a practical and unified manner, in the hope that other teachers may find herein some ideas useful to their students. Readers acquainted with the vast and deep complexities of this subject will recognize the necessity, in order to cover the subject usefully in relatively small space, to concentrate on a single, self-consistent viewpoint. They should not be misled, therefore, by the absence of adequate treatment of alternative views. References to a few of the pertinent recent papers in THIS JOURNAL (*1–9*) are cited to help make up for this deficiency, and some more general references are also appended.

This discussion ought logically to begin with a definition of a chemical bond. Let a bond be specified as an attractive interaction between two adjacent atoms. A bond is considered to exist when two adjacent atoms are so located that the potential energy is at a minimum, and when appreciable energy must be supplied to cause the atoms to separate. This latter energy measures the bond strength. All degrees of bond strength from zero to about 225 kilocalories (required to break Avogadro's number of bonds in nitrogen or carbon monoxide molecules) have been observed. An arbitrary choice of a lower limit of strength must be made, lest one include as bonds interactions so weak that the atomic clusters formed are too unstable to be dealt with experimentally as units. For practical purposes, let us establish the lower limit as 3 kcal per Avogadro's number of bonds. This includes all interatomic attractions customarily considered to be chemical bonds, even most "hydrogen bonds" (which are relatively weak), and excludes the weaker interactions known as van der Waals forces.

To begin to understand bonds, one must first have knowledge of the electronic structures of atoms. Atomic structure is described in many readily available sources, and general familiarity with it is assumed herein. The subject of bonding will therefore be treated directly, through consecutive consideration of a set of principles.

(1) **No atom can form a chemical bond if it has eight electrons in its outermost shell, or two electrons in its outermost shell if this is also the first shell.**

This applies to helium, with 2 outer shell electrons, and to neon, argon, krypton, xenon, and radon, each with 8 outer shell electrons. All these elements exist as monatomic gases having very low interatomic attraction and no capacity for bonding. They may, however, be trapped within cage-like structures of other atoms or molecules to which they form no bonds, in the so-called "clathrate" compounds. Evidently the structure uniquely characteristic of the inert atoms effectively shields the nucleus from attractive interactions with outside electrons, yet permits it to hold the atom's own electrons with exceptional strength.

(2) **All atoms can form chemical bonds if they have fewer than eight electrons in the outermost shell, or fewer than two electrons in the outermost shell if this is also the first shell.**

In other words, atoms of all the chemical elements except the six named above can form bonds and, therefore, chemical compounds.

(3) **Bonds are formed by the mutual sharing of electrons by two or more atoms, the result of electrostatic attraction between the shared electrons and the nuclei of the atoms.**

When two atoms approach one another, their interactions may be classified as threefold: repulsions between the two electron clouds, repulsions between the two nuclei, and attractions between the nucleus of each and the electrons of the other. Gravitational and magnetic effects are too small to have much significance, leaving to electrostatic attractions between unlike charges the major responsibility for bonding.

(4) **Mutual sharing of electrons can occur only when each atom has available one or more low energy orbitals capable of stably accommodating the shared electrons.**

The term "low energy orbital" means an orbital accessible to electrons from other atoms and permitting these electrons to come sufficiently within the influence of the nucleus that they can be stably held. In the "inert-shell" and "18-shell" type elements, these orbitals are primarily the outer shell *s* and *p* orbitals; sometimes outer *d* orbitals are also available for forming stable bonds. In the transition metals, underlying *d* orbitals are added to the orbitals mentioned for the other types of elements. Indeed, it is the special characteristics of these *d* orbitals that differentiate the chemistry of transition metals.

The requirement of electron vacancies in low energy orbitals suggests that electrons from other atoms cannot become closely enough associated with the nucleus to permit significant attractive interaction unless they can be accommodated in definite energy regions not already filled by electrons originally belonging to the atom. In turn, this suggests that a vacancy represents a vulnerable spot in an electronic armor which elsewhere tends to shield the nucleus from the influence of an electron from another atom.

(5) **Chemical bonding tends to use all available low energy orbitals, for attainment of maximum stability of the combination.**

This principle states the reasonable fact that since the attractions among atoms result from the sharing of electrons, more sharing results in greater attractions; therefore greater energy is necessary to separate the atoms and greater stability results. If a pair of electrons is brought from infinity and thrust upon an atom which initially attracts them, energy must ultimately be supplied to force the atom to accept them. For example, even an oxide ion cannot be formed stably through acceptance of an electron pair by an oxygen atom, though oxygen is second highest of all elements in electronegativity or attraction for electrons. It appears that as these outside electrons come into closer relationship to the nucleus, simultaneously they encounter greater repulsion from other electrons already present on the atom. Consequently the greater part of the stability potentially available is gained during the initial part of the process of acquiring the electrons. For this reason, it is usually more stable for a pair of electrons to be held by more than one atom than just by one, if orbitals are available on both. Or, in general, the more completely the available orbitals can participate in the bonding, the more stable is the molecule or ion.

(6) **When in each kind of atom involved, the number of vacancies exceeds the number of outer shell electrons, the shared electrons cannot use all available low energy orbitals by becoming localized between specific pairs of atoms, and therefore become relatively nonlocalized. The metallic state results, in which the atoms are closely packed with all the bonding electrons shared mutually by all the atoms.**

In all the elements of Groups IA, IIA, IIB, and IIIA of the periodic table, considering first only *s* and *p* orbital electrons and vacancies, the number of vacancies exceeds the number of electrons in the outermost shell. With the exception of boron, which is of an intermediate nature, all these elements are metals. Including boron, all in the solid state, whether as pure elements or mixed with one another, form crystalline lattices in which the atoms are closely packed, having far more neighbors than shareable electrons. Some of the elements having more *s* and *p* electrons than vacancies in the *s* and *p* orbitals can make use of outer *d* orbitals and thus also have an excess of vacancies; these, too, are metallic or metalloid: e.g., germanium, tin, lead, antimony, bismuth, tellurium. Apparently in metallic crystals, specific electrons are not shared between specific atoms, but rather, the outermost electrons are relatively nonlocalized and may be thought of as belonging to the metal atoms jointly. The shared electrons thus serve as a kind of electrostatic cement to hold the positively charged metal ions together. All the transition metals, of course, have an excess of available vacancies over shareable electrons. The bonding in metals is called "metallic bonding" and is characterized by the mobility and nonlocalization of the valence electrons (*6*).

Certain nonmetals, among them hydrogen, carbon, boron, nitrogen, and phosphorus, appear capable of entering into the lattice of certain metals, most notably transition metals, in nonstoichiometric proportions, without destroying the essentially metallic qualities of the crystal. In other cases, stoichiometric compositions result. Sometimes these compounds too have metallic properties and sometimes they do not, but seldom do they resemble closely the covalent compounds to be discussed below. Much remains to be learned concerning the nature of bonding in the solid state.

(7) **When the number of vacancies in at least one of the atoms involved does not exceed its number of outer shell electrons, the shared electrons tend to become localized between specific pairs of atoms.**

This is covalence. Here the shared electrons are not as mobile as in the metallic state, and consequently the properties of the aggregate are in general not very similar to the properties of a metal. Although normally covalence requires at least one atom having as many or more outer electrons than vacancies, covalent bonds may form among metal atoms in the vapor state, wherein, for example, the alkali metals are observed to be, to a limited extent, dimeric.

(8) **In bonding wherein electrons are localized, they are shared in pairs, thus involving for each bond formed, a total of two electrons, and one orbital of each atom.**

The maximum number of covalent bonds that can be formed is therefore limited to the number of electron pairs available for sharing, and limited by the number of orbitals available for the sharing.

(9) **One pair of mutually shared electrons suffices for a single bond, but the same pair of atoms may also share two pairs or three pairs of electrons, forming double or triple bonds.**

A bond formed by the sharing of one pair of electrons is called a single covalent bond. Double bonds are stronger and shorter; triple bonds are stronger and shorter yet. For example, two electrons are shared by the two carbon atoms in ethane, forming a single covalent bond with length (distance between the carbon nuclei) of 1.54 Å. Four electrons are shared by the two carbon atoms in ethylene, forming a double covalent bond with length of 1.35 Å. Six electrons are shared by the two carbon atoms in acetylene, forming a triple bond with length 1.20 Å.

(10) **Each pair of electrons shared between two atoms may consist, in covalence, of an electron from each atom, or, in coordinate covalence, of both electrons from one atom and none from the other.**

The simplest example of a covalent bond is to be found in the hydrogen molecule, H_2. Each hydrogen atom has one electron and one vacancy—an unpaired electron with its accompanying vacancy are the prerequisites for one covalent bond. Two hydrogen atoms unite by such a bond to form the hydrogen molecule, in which the two electrons are held jointly by the two nuclei. Similarly, a carbon atom has four electrons and four vacancies in its outermost shell, and thus can form four covalent bonds. The number of covalent bonds an atom can form is equal to the number of electron-plus-vacancy combinations it has available. With relatively few exceptions, in chemical reaction an atom forms as many single bonds (or their equivalent

in multiple bonds) as permitted by the number of electron-plus-vacancy combinations possible. This applies mainly to bonds using *s* and *p* orbitals; the number of bonds likely to be formed by use of *d* orbitals depends on conditions, and the number of electron-plus-vacancy combinations possible only sets an upper limit.

The "most stable state" of any atom implies filling the outer *s* orbital with its two electrons before adding any electron to an outer *p* orbital, but usually one of these *s* electrons is so easily promoted to a *p* orbital if one remains otherwise vacant, that the pairing of *s* electrons in the ground state of the atom does not limit the bonding ability of the atom in the valence state. For example, the electronic configuration of beryllium includes a pair of *s* electrons in the second and outermost principal quantum shell, with the three *p* orbitals all vacant. In this state, formation of ordinary covalent bonds is impossible, but beryllium readily forms two such bonds per atom. One of the *s* electrons is promoted to an otherwise vacant *p* orbital, the two valence orbitals become equalized (hybridized) and two "*sp*" orbitals, each containing one electron, are available for covalent bond formation. In all atoms having fewer than five outermost electrons, the ground state has at least one vacant *p* orbital and a filled *s* orbital if there are at least two electrons. In the valence state, however, one of the *s* electrons is promoted to an otherwise vacant *p* orbital and the orbitals become equalized (hybridized). One very familiar example is the carbon atom, which instead of leaving its ground state pair of *s* electrons intact in bonding, forms four identical sp^3 hybrid orbitals, each containing one electron. In the valence state of atoms, outermost electrons are seldom paired until all available orbitals are at least half filled (one electron in each orbital).

Exceptions to this promotion of an *s* electron are to be found in certain of the elements of higher atomic number, in which the *s* pair tends to remain intact and is therefore referred to as the "inert pair." This leads to compounds in which the combining power of these elements appears to be less than expected, by two. Thallium forms one bond in preference to three, mercury seems quite reluctant to form bonds at all, tin can about equally well form two bonds or four, and lead prefers two bonds to four. These generalizations hold mainly when the other component of the compound tends to withdraw electrons. When its attraction for electrons is more nearly that of the metal, then four bonds seem to be formed in preference by lead, three by thallium, and two by mercury.

When one atom supplies both electrons, and the other only supplies a vacant orbital, the bond is also covalent but has the special name, "coordinate covalent," or is called a "coordination bond." Atoms do not form this type of bond until they have first formed all the stable ordinary covalent bonds possible. For example, boron atoms in the valence state have three electron-plus-vacancy combinations and one vacant orbital. This orbital is not used in bonding unless the three normal covalent bonds or their equivalent have first been formed. Similarly, nitrogen atoms have also three electron-plus-vacancy combinations, but one filled orbital in addition. This electron pair is not used in bonding unless the three normal covalent bonds or their equivalent have first been formed. However, boron compounds, in which each boron atom is involved in three single covalent bonds, easily become further involved in coordinate covalence, making use of the fourth orbital. Nitrogen compounds, likewise, in which each nitrogen atom is involved in three single covalent bonds, easily become further involved in coordinate covalence, utilizing the extra pair of electrons with its orbital. When a boron compound such as BF_3 acquires a fluoride ion through coordination, all four fluorine atoms in the resulting complex BF_4^- ion are equivalent. Similarly, when ammonia coordinates a proton, forming an ammonium ion, NH_4^+, the coordination bond once formed is no longer distinguishable from the others, and all four hydrogen atoms are equivalent.

Simple positive ions always possess vacant low energy orbitals, and can thus act as acceptors to any available electron pairs on donor atoms. Thus result thousands of complex ions and coordination compounds (*3*, *5*, *9*).

(11) **When the shared electrons are localized in the bonds, different pairs of electrons, whether involved in the bonding or not, tend to become separated from one another as far as possible, which results in specific and predictable angles between two or more bonds to the same atom.**

Two covalent bonds can be formed by an atom having only two outermost electrons as well as by an atom having six outermost electrons. In two bonds to an atom of the first type, all outer electrons are used in the bonding. The farthest apart two electron pairs can be, on the surface of a sphere, is directly opposite one another. The bond angle is therefore 180°, this portion of the molecule being linear. All molecules in which two covalent bonds are formed by an atom of any IIA or IIB element have this linear orientation. When two single bonds to the second electronic type of atom mentioned above are formed, two pairs of electrons remaining unused in the bonding (*4*) bring the total, counting the bonding pairs, to four. The greatest possible separation of four positions on the surface of a sphere is at the corners of a regular tetrahedron, resulting in bond angles of 109° 28′. With (usually) only relatively minor deviations, as discussed below, this is the angle between most pairs of single bonds formed by VIA elements.

Three covalent bonds can be formed by an atom having only three outermost electrons as well as by an atom having five outermost electrons. In the first type, all outer electrons are used in the three covalent bonds. When these three electron pairs have separated as far as possible, they form an equilateral triangle with the nucleus at its center. Bond angles are therefore 120°. On the other hand, in the second type of atom where an extra pair of electrons remains after the three covalent bonds are formed, the total of four pairs must again be distributed tetrahedrally. Bond angles approach 109°, and with three single bonds, the structure is that of a low pyramid, with an unused electron pair occupying a fourth tetrahedral position above its apex, instead of planar as when the fourth orbital is vacant.

Atoms having four outermost electrons and four

vacancies naturally form four bonds which are tetrahedral, with bond angles of 109° 28′.

Formation of more than four bonds leads similarly to distribution of electron pairs at their widest possible separation. With five electron pairs all involved in five bonds, a trigonal bipyramid results, and with five electron pairs but fewer than five single bonds, the structures observed can usually be explained as trigonal bipyramidal with one or two positions occupied by unshared electron pairs. With six electron pair bonds, the greatest possible separation results in a regular octahedral structure, with bond angles of 90°.

The same practical principle applies when one or more of the bonds is multiple. For example, when all outer electrons of carbon are used in two bonds, as in CO_2 (two double bonds) or acetylene (one single, one triple bond), the bond angle is 180°. When all outer electrons are used in three bonds, as in ethylene and benzene carbon atoms, the bond angles are close to 120°.

Deviations from the expected angles are common but usually not very great, and usually easy to explain in a qualitative way. Two multiple bonds tend to repel one another more strongly than two single or one single and a multiple bond. The nitrogen bond angles in HNO_3, for example, are 130° between the two "lone oxygens" and 115° between each of them and the hydroxyl oxygen, instead of 120° for all three. Outer unshared pairs of electrons appear to repel one another more than bonding pairs; two unshared pairs thus tend to force two bonding pairs closer together than otherwise. This may help to explain, for example, why the bond angle in water is 104.5° instead of 109.5°. Also, the use of underlying *d* orbitals causes some irregularity in structure of some compounds; $TiCl_4$, for example, may not be quite regularly tetrahedral. In fact, all bond angles have been explained in terms of orbital types and hybridization, but the above account seems at least equally successful, and has the advantage of being much more easily understood. (See also *1*, *8*).

(12) **Electrons are most stably shared when they can be concentrated directly between the two nuclei and as far as possible from other outer electron pairs.**

This occurs in single covalent bonds, unless prevented by a molecular geometry which causes distortion of bond angles. For example, the sharing of electrons produces stable bonding in ethane but in cyclopropane the closure of the ring requires decreasing the bond angle from the normal 109° 28′ to 60°. This must result either in the bonding electrons not being concentrated between the carbon atoms as directly as otherwise possible, or in bonding electron pairs being closer together than otherwise necessary. By either interpretation, the bonds are made less stable. Another example is white phosphorus, whose P_4 molecules require 60° bond angles instead of more nearly tetrahedral. White phosphorus is consequently much less stable and more reactive than other forms of the element wherein the electrons can be more stably shared.

Electrons that are in addition to the first, single bonding, pair cannot be concentrated between the bonded atoms because of the repulsion by the first pair. Thus in all multiple bonding, the electrons supplementing the initial pair are in orbitals directed obliquely rather than directly toward the other atom, and in general must therefore be less effective in the bonding. With a few important exceptions (such as N_2, CO_2, etc.) this leads to lower added stability per electron, for each electron added to the bond, so that double bonds are not twice as strong as single bonds, nor triple bonds three times as strong.

Both causes of reduced stability of sharing tend to lead to addition polymerization, by which either the angle strain is removed or the extra electrons of multiple bonds become more effectively used in additional single bonds. For example, ethylene oxide forms polyethers and ethylene forms polyethylene.

(13) **When the conditions for covalence are met but the localization of shared electrons between specific pairs of atoms does not make use of all available vacancies, either outer electrons otherwise uninvolved in the bonding tend to become shared too (using the otherwise unoccupied vacancies), or the electrons already used in the bonding become less localized by extending to the otherwise unoccupied vacancies.**

This principle may be regarded as an extension of principle (6), which describes metallic bonding as making use of all available vacancies despite a deficiency of electrons for ordinary covalence. The difference is that principle (6) refers only to atoms whose number of outer electrons is less than the number of vacancies, whereas here at least one atom in the compound has as many or more outer electrons as vacancies.

Examples of the use of otherwise vacant orbitals to increase the electron sharing are especially common in compounds wherein the IIIA elements, whose atoms have four outer orbitals but only three electrons, are joined by three single bonds per atom to other elements (*2*). Certain boron compounds, for instance, appear to contain three single covalent bonds to fluorine, or to oxygen, but the bond lengths are somewhat less than predictable on the basis of polarity alone, which does result in shorter bonds. Evidently an otherwise unshared electron pair on the fluorine or oxygen can interact with the boron through the use of the fourth and otherwise vacant orbital of the boron, thus strengthening the bond. This will be discussed further under principle (14); see B of Figure 1.

Aluminum chloride is another well-known example. Instead of remaining $AlCl_3$, this molecule dimerizes to Al_2Cl_6 in vapor, in solution in nonpolar solvents, and presumably in the liquid state. An otherwise unshared pair of electrons on one chlorine on each aluminum is attracted to the other aluminum through its fourth and otherwise unoccupied orbital. The two $AlCl_3$ units are thus bridged by two of the chlorine atoms.

Examples of the extension of electrons already used in bonding to otherwise unoccupied vacancies are presented by the boron hydrides and related compounds, and by certain organo-metallic compounds. For instance, the simplest compound of boron with hydrogen would be expected to have the formula BH_3. Here all electron pairs are used in the three bonds, yet a fourth orbital of the boron is unoccupied. Two BH_3 units combine, the true molecule being B_2H_6. Evidently an electron pair on each boron,

which is already used in the bond to one hydrogen atom, spreads out to occupy the otherwise vacant orbital of the other boron as well. Two hydrogen atoms thus become equally held by the two boron atoms, each bridge using only two electrons.

Similarly, aluminum trimethyl has the molecular formula, $Al_2(CH_3)_6$. Here the two aluminum atoms are believed to be bridged by two of the methyl groups, or perhaps more specifically, by their carbon atoms. The bridging methyl groups use the electron pair normally localized between carbon and aluminum, to occupy partially an otherwise vacant orbital on the other aluminum, so that the same pair of electrons occupies an orbital of the carbon and simultaneously an orbital of each aluminum.

(14) **When an atom possesses an otherwise vacant orbital that might equally stably (or nearly so) accommodate an electron pair from any one of two or more neighboring atoms, this orbital tends to make use of all possibilities partially, rather than one exclusively.**

(15) **Similarly, when two or more atoms each possess a vacant orbital and the same pair of electrons is equally available, with equal stability or nearly so, to each orbital, the electron pair tends to occupy not one exclusively but all equally.**

Together, these two principles deal with situations that cannot be described adequately in terms of electron pair bonding. Such situations are commonly referred to as involving "resonance" (7), which in this application is an attempt to describe nonintegral electron bonding as something intermediate among definite electron-pair forms.

The concept of resonance requires that one try to visualize the electronic configuration of a molecule as a "resonance hybrid," an intermediate between configurations that represent conventional single and multiple covalence. By Wheland's well-known analogy, a mule is a "resonance hybrid" of a horse and a donkey, bearing some resemblance to each but being neither one. It is hardly satisfying to be limited to one's acquaintance with horses and donkeys in attempting to picture an animal which is neither. Far better if one could picture the mule as it is, thus making a knowledge of horses and donkeys unessential, and avoiding the possibility that such knowledge may lead to fallacious inferences concerning the special qualities of mules. An attempt is therefore made here to develop a visualization of ions and molecules in which resonance is said to occur, as they actually are, rather than in the form of nonexistent "contributing structures." Unfortunately, this is not always easy to do.

An example classifiable under principle (14) is the nitrate ion. This has a planar structure, suggesting that all valence electrons are involved in the three N—O bonds, for if a pair were left over, it would force the three bonds toward the corners of a tetrahedron around the nitrogen, and a pyramidal arrangement would result. The three bonds, moreover, are experimentally indistinguishable from one another. The problem of interpretation in terms of ordinary covalent bonds arises from the fact that the three bonds must involve six electrons on the nitrogen (the extra electron of the ion plus the original five) but also four orbitals. The fourth orbital cannot be involved if three single bonds occur. It could become involved with a pair of oxygen electrons, forming a double bond with one oxygen, but this would mean one double bond and two, longer, single bonds. Furthermore, no one of the three oxygen atoms can contribute an otherwise unshared electron pair more easily than any other.

One may well ask here, why must this fourth orbital become involved at all? Remember that a vacant low energy orbital represents a soft spot in the armor of electron shielding of the nucleus, a point at which the nuclear charge can effectively exert its attraction for "foreign" electrons. Whenever possible, then, such an orbital is used in bonding instead of lying vacant, and a more stable molecule or ion results.

The resonance interpretation of the nitrate ion is, in this example, relatively simply represented as in A of Figure 1.

An alternative view proposed here is that this ion may be regarded as containing single covalent bonds, and superimposed upon this structure is the otherwise vacant nitrogen orbital, which may accommodate a pair of otherwise nonbonding electrons of an oxygen atom. The fundamental principle of resonance, as suggested above, is that whenever more than one practically equivalent arrangement of electrons is possible, greater stability results from an average or intermediate arrangement, or from using all available electron pairs even if only partially, rather than leave any unused. As a result, the fourth orbital of the nitrogen partially accommodates a pair (or each of the

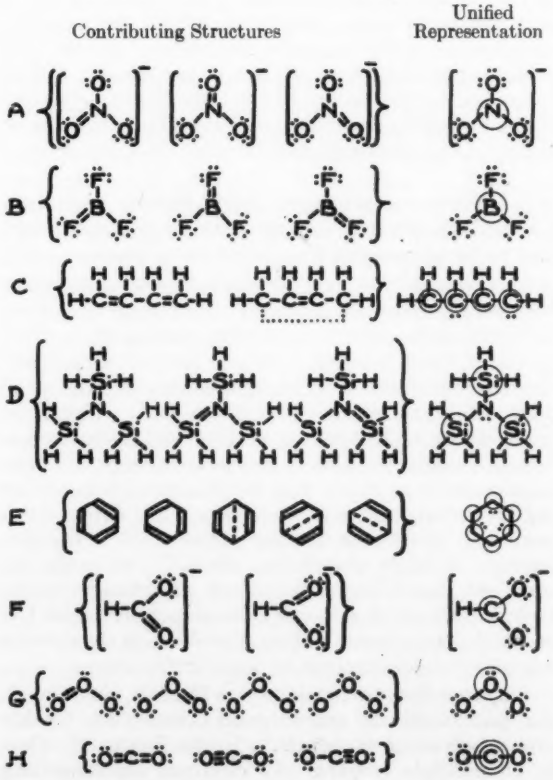


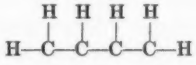
Figure 1. Representation of resonance structures.

available pairs) of electrons from each oxygen instead of fully accommodating only one pair from one oxygen. If this fourth orbital is represented by a circle around the nitrogen, the nitrate ion can be represented by the single formula shown in A of Figure 1.

Similarly, the boron compounds cited under principle (11) have the single otherwise unoccupied orbital per boron atom that might accommodate a pair of electrons from any one of three fluorine or three oxygen atoms. The experimental evidence indicates that the actual bonds are alike, suggesting that if they are partly multiple, they must be equally so. The boron trifluoride molecule, for example, might be represented as a single-bonded structure, superimposed upon which is the fourth boron orbital which can make partial use of otherwise unshared electron pairs of all three fluorine atoms. The "contributing structures" and "unified representation" are shown in B of Figure 1.

In general, the scheme proposed here for representing "hybrid" structures is to write down the conventional structural formula using only single bonds to connect the atoms. Each otherwise vacant orbital that would be expected to participate in the bonding if possible is then represented as a circle around the atomic symbol (two concentric circles if two orbitals are so available, as in CO_2, H of Fig. 1). Electron pairs that are not shared in the single bonding but might participate in the bonding in conjunction with the "extra" orbitals are drawn in as the usual pairs of dots. Where it is desirable to assign these electron pairs to an atom in which an extra orbital is already indicated, the dots are placed outside the circle that represents the orbital, as in benzene (E of Fig. 1). When, however, the electron pair is believed to remain uninvolved in the bonding, it is placed inside the circle, as on the central oxygen of ozone (G of Fig. 1). One understands from such a representation that a vacant orbital may become involved (to some extent, often equally) with all pairs of electrons on adjacent atoms that are available to it. Similarly, an electron pair may become involved (to some extent, often equally) with all available orbitals on adjacent atoms. Further examples are discussed in the text following, and shown in Figure 1.

A familiar organic example of resonance is 1,3-butadiene (7). From the viewpoint under consideration here, this can be regarded as basically a single-bonded structure:



In this structure, each carbon atom forms only three single bonds, thus leaving a total of four vacant orbitals, and four electrons, or two pairs, uninvolved in the single bonding. These orbitals and electron pairs might add to the single bonded structure in two ways. If the electron pairs become associated one with each of the two terminal pairs of carbon atoms, then all four orbitals are used in two double bonds, giving the usual structure. Alternatively, one pair of electrons might occupy jointly the two orbitals of the central pair of carbon atoms, forming a double bond here. The second pair of electrons would then occupy an orbital of one terminal carbon. Barring ring closure, which would introduce bond angle distortion and therefore result in reduced stability, the second arrangement is not as stable as the first. However, the familiar 1-4 addition to butadiene, and the observed shortening of the 2-3 bond and lengthening of the 1-2 and 3-4 bonds over expectations, suggest that the second arrangement is a contributing characteristic of the normal butadiene molecule, and perhaps becomes the chief characteristic under the influence of certain attacking reagents. A satisfactory single representation of the molecule would be that of C, in Figure 1. The electron pair shown on carbon 2 can move either to the left or to the right. If it moves to the left, the pair on carbon 4 can also move left and the familiar structure results. If the pair on carbon 2 moves to the right, however, the pair on carbon 4 is trapped where it is.

Principle (15) is illustrated by the compound, trisilylamine, $(SiH_3)_3N$, which, contrary to initial expectations, has a planar rather than pyramidal structure and is not an electron donor. Here the nitrogen atom has a pair of electrons that would ordinarily be unshared, except when the molecule acts as a base. On the three silicon atoms, however, although the four *s* and *p* orbitals are used in the bonding, outer *d* orbitals are available that might receive electrons. No one silicon atom could hold an electron pair more strongly than any other; consequently, all three hold the electron pair equally. The resulting structure might be represented in a single diagram, as shown in D of Figure 1.

(16) **In a cyclic system where each ring atom might equally well form a double bond to one ring neighbor and a single bond to the other, or vice versa, but not double bonds to both at once, alternating bonds do not form if a planar configuration permits the multiplicity to even out over all the ring bonds.**

The simplest example of this is, of course, benzene, C_6H_6. This may be regarded as basically a single-bonded structure, each carbon atom being joined to three other atoms, one hydrogen and two carbons. This leaves six electrons, one from each carbon, and six orbitals, one on each carbon, uninvolved in the bonding. Six electrons, or three pairs, will tend to occupy the maximum number of orbitals. The best arrangement will therefore be in three additional single bonds, where each pair of electrons occupies two orbitals, using in all, the six orbitals that are available. These, superimposed upon the single bonds already present, make three double bonds and leave three single bonds. Here, however, exactly equivalent arrangements, the well-known Kekulé structures, are possible. In keeping with the principle of resonance, neither structure occurs, but instead, the fourth orbital of each carbon atom becomes involved equally in partial bonding to both carbon neighbors, as represented in E of Figure 1.

(17) **Electrons which through equalization of all ring bonds of a planar covalent ring molecule are relatively nonlocalized, can be shared with metal atoms, forming bonds that join the ring as a whole, not through one particular atom, to the metal atom.**

Ferrocene, benzene chromium compounds, and similar substances illustrate this principle, for in them, the metal atom appears to form bonds to the ring as a unit, the nucleus attracting the relatively mobile "pi" electrons.

Somewhat similar are metal-olefin complexes, in which olefin molecules act as ligands, attaching to metal ions not through an individual carbon atom but through the agency of the double bond itself acting as electron donor.

(18) **Bonding electrons commonly are unevenly shared, because atoms of the different elements differ in their attraction for electrons, called "electronegativity."**

Electronegativity appears to be a fundamental quality rather than quantity and can only be derived on a relative basis. However, numerous and widely different approaches have produced values in reasonably good agreement, and for only a few elements are the differences significantly controversial. Bonding electrons are believed to be shared among the atoms in proportion to their initial electronegativities. Some elements in order of increasing electronegativity are: Cs, Rb, K, Na, Li, Ba, Sr, Ca, Mg, Be, Al, Cd, Si, B, Zn, In, Hg, Tl, Pb, Sn, Bi, P, Sb, H, Ge, Te, C, I, As, S, Se, N, Br, Cl, O, and F.

(19) **Uneven sharing produces partial negative charge on the atom having greater than half share, leaving partial positive charge on the atom having less than half share, and making the bond polar.**

Uneven sharing means that on a time-average basis, the shared electron pair is more closely associated with one nucleus than with the other. Consequently the effect is of a surplus of negative charge around the one nucleus and a deficiency around the other. A deficiency of negative charge, since the nuclear charge remains unchanged, is equivalent to an effective positive charge. The greater the electronegativity difference, the greater the unevenness of sharing, and of course the greater share is acquired by the initially more electronegative atom.

Uneven sharing makes the bond polar. If there is but one bond in the molecule, or unless symmetrically compensated for by the polarity of other bonds in the molecule, bond polarity will cause molecular polarity, and is one of several factors influencing the over-all electrical distribution in a molecule that is measured as the "dipole moment."

(20) **For each atom of a compound or ion, the "partial charge" expected from initial electronegativity differences can be estimated quantitatively, and applied usefully as an index of the condition of the combined atom.**

A simple method is available for determining quantitatively the relative polarity of bonds and indicating the condition of each combined atom. Briefly, this method depends on estimating the change in electronegativity that would correspond to gain or loss of one electron. The ratio of the change in electronegativity undergone by an atom in forming a compound, to the electronegativity change it would have undergone had it acquired unit charge, is taken as the partial charge on the combined atom.

Partial charge is not to be taken literally, but rather as an index of the condition of a combined atom. A knowledge of this condition not only is invaluable in chemical interpretations, but also contributes to an understanding of bonds, as follows:

(*a*) Atoms tend to be better electron donors with increasing negative charge, and lose donating power with diminishing negative and increasing positive charge. The fluorine atoms in BF_3 may be regarded as acting as electron donors toward the boron, but cannot do so nearly as well as the fluorine ion, which therefore becomes attached to the boron forming the fluoroborate ion, BF_4^-. Combined nitrogen commonly acts as electron donor, as in ammonia and amines, but therein it bears negative charge. When its charge is positive, as in NF_3 and $N(CF_3)_3$, its electron-donating properties are lost.

The ability of oxyacid anions to donate electrons to protons, which is inversely a measure of the acid strength of the resulting oxyacid, is found to be closely related to the partial charge on oxygen in the anion; the higher the negative charge, the better the donor.

(*b*) Atoms tend to be more effective electron acceptors, the higher their positive charge. For example, etherates of boron halides, in which the charge on boron is relatively high, are much more stable than etherates of BH_3 and boron trimethyl, in which the charge on boron is relatively low.

(*c*) Bonds between atoms both positively charged tend to be weakened, supposedly by relative unavailability of the bonding electrons. Oxalic acid, in which the two adjacent carbon atoms bear positive charge, is relatively unstable. In the higher silanes, silicon-silicon bonds appear to be unexpectedly weak, but each silicon bears positive charge. Phosphorous acid, H_2HPO_3, in which one positively charged hydrogen atom is attached directly to a positively charged phosphorus atom, is quite unstable and tends to disproportionate to phosphine and phosphoric acid, in neither of which are adjacent atoms positively charged. (Important exceptions are the fluorocarbons and, to a lesser extent, the chlorosilicons.)

(*d*) Other factors being equal or negligible, bond strength tends to increase with increasing polarity. For example, heats of formation per equivalent for any series of halides of the same element in increasing positive oxidation states seem always to diminish.

(*e*) The existence of protonic bridging appears to depend on the presence of partial positive charge on the hydrogen and partial negative charge on the atom of the other molecule to which the bridge is made. Hydridic bridging appears never to occur unless the hydrogen bears partial negative charge, and the bridged atoms partial positive charge. Alkyl group bridging similarly seems to depend on at least a minimum negative charge on the carbon and partial positive charges on the bridged atoms.

(21) **When the formation of small individual molecules, by utilization of all available electron-plus-vacancy combinations on each of the combining atoms, would leave exposed both vacant orbitals and outer electron pairs, such molecules form only in the vapor phase, if at all. At ordinary temperatures, crystalline solids result, in which no individual molecules can be distinguished, and no outer orbitals and electron pairs (except on the crystal surface) are left exposed.**

Common examples of this principle, which is an extension of principle (5), are given by the binary compounds of metals with nonmetals. Only when geometrical symmetry prevents exposure of vacant orbitals

are these compounds molecular, as for example, Al_2Br_6, WF_6, OsO_4, and Mn_2O_7. Otherwise they are nonmolecular crystalline solids.

In all such solids, each metal atom is surrounded by more nonmetal atoms, and each nonmetal atom by more metal atoms, than could unite with it by ordinary covalence. Furthermore, the adjacent neighbors of any given atom are usually equidistant from it, indicating that all are bonded equally to it. The problem is to recognize and describe the nature of this bonding.

For example, isolated atoms of sodium, each able to form one covalent bond, can unite with isolated atoms of chlorine, each also able to form one covalent bond, to produce isolated molecules of NaCl. In these molecules, the electron sharing is very uneven because chlorine initially is much more electronegative than sodium. Therefore it gains much more than half share of the bonding electrons, enough to give it an estimated charge of -0.67 (electron), leaving a charge of 0.67 on the sodium. The bond is thus highly polar.

If the chlorine atom could capture complete control of the shared electrons, and thereby reach the limit of maximum polarity of the covalent bond, a chloride ion, Cl^-, would result, as well as a sodium ion, Na^+. An electrostatic attraction between the oppositely charged ions would then constitute the bonding force. This is "electrovalence," or "ionic bonding." Covalent bonding and ionic bonding are conventionally taught as distinct types of bonding, and then most textbook authors agree that most bonds fall somewhere between these extremes of even sharing and complete electron transfer.

Indeed a real question exists as to whether an isolated molecule of NaCl actually contains a highly polar covalent bond, as indicated above, or consists of an ion pair. Various interpretations of experimental evidence bearing on this question are not in agreement. But more important is the fact that, whatever isolated NaCl molecules may be, they exist only in the vapor state, which requires high temperatures. Cooling below 800° produces the familiar crystalline salt, in which an atom of each element is at the center of a regular octahedral sextet of atoms of the other.

The familiar accepted interpretation of this structure is that this is simply an aggregation of ions, charged particles whose fields are symmetrical about the nucleus and therefore equally effective in all directions. The particular structure observed is believed to be the most symmetrical possible arrangement of the component particles that could result, consistent with their relative sizes and the facts that the like charged sodium ions repel one another, the like charged chloride ions repel one another, and the sodium and chloride ions, being of opposite charge, attract one another. Sodium chloride is classified as a typical "ionic solid."

It is helpful to recognize that each sodium ion has four outermost vacant orbitals, which are potentially electron acceptors, even if not strongly so. Each chloride ion has four outermost electron pairs, which potentially (by virtue of the excess of electronic charge) can be donated, even if not easily so. Surely the close proximity of these vacant orbitals and electron pairs in the crystal must result in some interaction. Perhaps the following discussion may shed some light on the nature and extent of such interaction.

A chloride ion has six sodium ion neighbors but only four outer electron pairs; this could be considered as corresponding to donation of $^2/_3$ of an electron pair to each adjacent sodium ion. Receiving $^2/_3$ of an electron pair from each of six chloride ions, a sodium ion would thus be surrounded by $6 \times {}^2/_3$ or 4 electron pairs. Thus each chlorine and each sodium share eight outer electrons and all available outer electrons and vacancies are used, giving greatest possible stability to the aggregate. Now if the reasonable assumption is made that in the solid (as well as in the molecule), bonding electrons become distributed among the sharing atoms in proportion to their initial relative electronegativities, then a charge of 0.67 on each sodium "ion" means it has 0.33 electron more than it should. But in the solid, 8 electrons are shared, and 0.33/8 is 4%. The bonding can then be described by saying that the bonding electrons are held 96% by the chlorine and 4% by the sodium. The important difference between the single molecule and the crystal is that in the former, only two electrons are shared, but in the crystal, eight.

In a similar manner, "fluorite," a crystalline compound, CaF_2, may be examined. The calculated charge on calcium is 0.94, which suggests that each calcium "ion" has 1.06 electron more than it should. In the fluorite structure, each fluoride ion has four calcium neighbors, and could provide one electron pair to each. Each calcium has eight fluoride ions around it, and *d* orbitals must be called into use if each donates an electron pair. Assuming this to be, then the total number of electrons surrounding each calcium is 16. Then 1.06/16 is 6.6%, which is calcium's share of the bonding electrons, leaving 93.4% as the share held by the fluorine.

The concept of the actual existence of simple ions in the solid state becomes strained in consideration of the so-called "ionic oxides" such as CaO. The crystal structure is essentially that of NaCl. Thus each "oxide ion" would be surrounded by six doubly positive calcium ions. Actually oxygen cannot take on two extra electrons per atom stably; energy must be supplied to force the formation of an oxide ion, O^{--}. Then how could it possibly exist in such an electron-attracting environment as that of six doubly charged cations? The calculated charge on calcium in calcium oxide is 0.57, which suggests that each "ion" of calcium has 1.43 electron more than it should. Then 1.43/8 is 18% share in the bonding electrons by calcium, leaving the oxygen to hold 82% of them.

Compounds such as those just described are good conductors of electricity, when in the molten state, by transport and discharge of ions; in some cases this also occurs in the solid. They can be solvated by polar solvents giving conducting solutions showing the solvated ions to have considerable freedom of movement. Such observations seem to support the concept of their structure as consisting essentially of simple aggregates of ions. Yet all degrees of "ionic character" of solids are believed to be possible, and many compounds, such as zinc sulfide, possess structure definitely indicating something other than a symmetrical packing of oppositely charged spheres. In zinc blende and wurtzite both, no individual ZnS molecules are distinguishable. The structure consists of each zinc atom surrounded

tetrahedrally by four sulfur atoms, and each sulfur atom by four zinc atoms. Yet the calculated charge on zinc is only 0.16. If each zinc atom shares 8 electrons with sulfur, and has 1.84 more electrons than the ion would have, then 1.84/8 or 23% is zinc's share of these electrons, leaving 77% to the sulfur. It should be noted that since six of the eight electrons originate with the sulfur, the zinc share would only be 25% if zinc were like sulfur in electronegativity.

Obviously much remains to be learned about bonding in the solid state, but at least the "ionic" and the "covalent" crystal lattices can be viewed on the common basis of a tendency to make fullest possible use of outer electrons and vacancies. In general, the more uneven the electron sharing, the more nearly the properties of the compound approach those commonly associated with "typically ionic" crystals.

When complex ions are involved, the association of oppositely charged ions may or may not involve elements of covalence. In the tetraalkyl ammonium hydroxides, for example, the hydroxide ions must be quite independent of the cations, except to be under the general influence of their net partial charge. Naturally, separation of the ions in water occurs readily and these compounds are strong bases. In calcium carbonate, on the other hand, each oxygen has a coordination number of three, being directly associated with two calcium atoms as well as the carbon, and each calcium atom is octahedrally surrounded by six oxygen atoms. The simplified picture of alternating calcium and carbonate ions appears quite inadequate here, for the structure strongly suggests that a pair of oxygen electrons is directly involved in each attachment to calcium.

(22) **Stable compounds can be formed between any positive and any negative ion, whether simple or complex, provided the size and shape of the ions permits the formation of a stable crystal lattice, and provided also that neither ion is made unduly unstable through polarization by the other.**

Combination of a positive and a negative ion will not necessarily produce an ionic substance, of course, if the positive ion is capable of removing charge from the negative ion. For example, combination of a hydrogen ion with a chloride ion produces molecular HCl. Also, many potentially ionic lattices would be unstable because of deformation, especially of the anion by the cation. For example, calcium carbonate is stable, but hydrogen carbonate and aluminum carbonate cannot be isolated owing, presumably, to the high polarizing power of the proton and the aluminum ion; for similar reasons, beryllium carbonate decomposes at about 0°. Contributing to this instability of many complex salts no doubt are other factors, such as the crystal energy of the decomposition products. When a complex anion is protected from the cation by coordination of ligands such as water molecules around the cation, stability of the ionic salt is more common; many hydrated salts (for example) exist, for which the anhydrous counterpart is unknown or much less stable.

(23) **When the electron of a hydrogen atom is partially withdrawn from it, the absence of other electrons to shield its nucleus permits electrostatic attraction between the nucleus and an electron pair of another atom that is frequently strong enough to be considered a bond.**

This is the "hydrogen bond." The only electrons around a hydrogen atom must be valence electrons. The partial withdrawal of these by a more electronegative atom leaves the hydrogen orbital less than fully occupied, and the hydrogen nucleus is thus exposed to an electron pair of an atom of another molecule. This other atom must be quite negative and quite small in order for its unshared electron pairs to be available to attract hydrogen strongly enough to form a bond. It must be quite negative in order that its electron pair can be more readily accessible. It must be quite small because, presumably, an electron pair is of necessity concentrated in a small atom but relatively free to spread out over the surface of a larger atom, such that a nearby positive hydrogen can interact less effectively. A relatively high negative charge means that the atom must originally have been high in electronegativity as well as small, which practically restricts its identity to fluorine, oxygen, or nitrogen. The hydrogen is also usually covalently attached to one of these atoms, although it can become sufficiently positive to form such bonds when it is bonded to carbon, if the other substituents have sufficient electron withdrawing power, as in CHF_3. In such bonding, the hydrogen atom is a bridge, sometimes within a molecule but more commonly, in ordinary chemistry, between two molecules. It does not become equally attached to both bridged atoms, but remains tightly attached to its original partner and considerably more distant from the more weakly attracted negative atom of the other molecule. The term "hydrogen bond" is most common; it is also called "hydrogen bridge" or "proton bond"; to distinguish it from hydridic bridging, it has been suggested that it be called "protonic bridging."

The bifluoride ion, FHF^-, commonly cited as an example of protonic bridging, seems more appropriately classed by itself, since here the bonding is several times stronger, the hydrogen is evenly located between the two fluorine atoms, and it bears a slight negative charge, if distribution of the extra electron among the component atoms in proportion to their electronegativities is assumed. It seems to demonstrate a tendency to make fuller use of the hydrogen orbital than is made in HF itself, wherein the fluorine holds much more than half share of the valence electrons.

(24) **Potential bonding capacity can be fully realized only if spatial requirements of participating atoms or groups can be met.**

In other words, two groups that are geometrically unable to fit together cannot form a bond, and if steric difficulties do not prevent bond formation altogether, they may weaken the bond.

(25) **Every bond is influenced, directly or indirectly, by every electron and every nucleus in the aggregate. The interactions producing this influence are so extremely complex that they will probably defy simple analysis forever.**

Statements such as these 25 must not be regarded as truly fundamental explanations. It is hoped they can be helpful as a qualitative description and a practical guide.

Summary

All chemical bonding may be thought of as a process of consolidating atoms into aggregates whose energy content, as the result of mutual sharing of electrons by the several nuclei, is substantially lower than the sum of the energies of the separate atoms. Mutual sharing of electrons can occur only when at least one atom of the two forming a bond has vacancies in outer low energy orbitals, for such vacancies are essential to effective attraction between the nucleus of that atom and electrons of the other atom. In general, chemical combination tends to continue until all exposed low energy electron vacancies are effectively occupied. Electron sharing ordinarily involves localization of a pair of electrons between a specific pair of atoms only when in at least one of these atoms, the number of outermost electrons equals or exceeds the number of outermost vacancies. The shared electrons are usually relatively nonlocalized in all other bonding. Whether localized or nonlocalized, outer electrons tend:

(*a*) to become distributed over as many atomic orbitals as possible;

(*b*) to become distributed among the atoms of a compound in proportion to the initial relative electronegativities of the atoms.

Both these tendencies are toward lower energy and thus greater stability.

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Textbook Errors, 32

The Equilibria of Complex Formation

The increased emphasis on the structural approach to solution chemistry which has come about in the past few years has led to a resurgence of interest in the formation and dissociation of complex ions. The treatment of the subject is begun early in the first course in chemistry. Despite the present recognition of the stepwise formation of complexes in solution, many textbooks[1] continue to treat it as a one-step process, presumably to preserve a reasonable simplicity in the mathematical computation of concentrations. Such concentrations can be in error by a large factor.

It is the purpose of this paper to review the equilibria briefly, point out the common error, and indicate how it can be corrected without unnecessarily complicating the equilibrium calculations. Calculations which involve conditions similar to those ordinarily encountered in the laboratory can be treated readily.

Formation and Dissociation of Complexes

In his classic study on metal ammine complexes Bjerrum (*1*) defines the following equilibria for a "step" system consisting of a central group M and N ligands A:

$$M + A \rightleftharpoons MA$$
$$MA + A \rightleftharpoons MA_2$$
$$MA_{N-1} + A \rightleftharpoons MA_N$$

These equilibria are found in acid-base, redox, and

Suggestions of material suitable for this column and guest columns suitable for publication directly are eagerly solicited. They should be sent with as many details as possible, and particularly with references to modern textbooks, to Karol J. Mysels, Department of Chemistry, University of Southern California, Los Angeles 7, California.

[1] Since the purpose of this column is to prevent the spread and continuation of errors discussed, and not the evaluation of individual texts, the source of errors discussed will not be cited. The error must occur in at least two independent standard books to be presented.

complex formation reactions. A complex may lose its ligands in the same manner as polyprotic acids lose their protons or reducing agents lose their electrons.

Bjerrum's investigations indicated that the formation of a coordinately saturated complex always takes place through all the theoretically possible intermediate steps.

For example, the formation of the diammine silver complex is represented by

$$Ag^+ + NH_3 \rightleftharpoons AgNH_3^+$$

$$k_1 = \frac{[AgNH_3^+]}{[Ag^+][NH_3]} = 2.1 \times 10^3$$

$$AgNH_3^+ + NH_3 \rightleftharpoons Ag(NH_3)_2^+$$

$$k_2 = \frac{[Ag(NH_3)_2^+]}{[AgNH_3^+][NH_3]} = 7.7 \times 10^3$$

The constants k_1 and k_2 are called formation constants and are numbered according to the number of ligands in the complex on the right-hand side of the chemical equation (*2*). The formation constants are the reciprocals of the instability constants which are often written.

Until the last few years few formation constants had been measured accurately. Therefore, the usual custom is to combine the equilibria as shown in this example

$$Ag(NH_3)_2^+ \rightleftharpoons Ag^+ + 2NH_3$$

$$K_{inst} = \frac{1}{k_1 k_2} = \frac{[Ag^+][NH_3]^2}{[Ag(NH_3)_2^+]} = 6.2 \times 10^{-8}$$

K_{inst} is termed the instability constant of the complex. Since it represents only an algebraic manipulation of the formation equilibria and constants, the instability constant expression is valid. Nonetheless, it must be clearly understood that the elimination of $[Ag(NH_3)^+]$ from the equilibrium expression does not mean that it does not exist in the solution. Quite to the contrary, its presence cannot be ignored when conservation of mass equations are applied to the system. The sum of the Ag^+ and $Ag(NH_3)_2^+$ concentrations is not equal to the total concentration of silver present. Neither is (NH_3) equal to $2[Ag^+]$ as a general rule, even if the solution is one of the complex alone.

A very common textbook problem asks that the silver ion concentration, $[Ag^+]$, be calculated when the pure complex ion is contained in water. Given only K_{inst} it is necessary to assume that $[NH_3] = 2[Ag^+]$ and to ignore the existence of the monammine silver ion, $AgNH_3)^+$. If one considers a solution of 0.0200 *M* Ag $(NH_3)_2^+$, he finds $[Ag^+] = 6.8 \times 10^{-4}$ *M*. A more precise value can be calculated by solving a group of four simultaneous equations which make use of the formation constants k_1 and k_2. This process yields $[Ag^+] = 3.2 \times 10^{-4}$ *M* and $[NH_3] = 1.9 \times 10^{-3}$ *M*. (Also $[AgNH_3^+] = 1.3 \times 10^{-3}$ *M* and $[Ag(NH_3)_2^+] = 1.9 \times 10^{-2}$ *M*.) The silver ion concentration is seen to be somewhat less than one-fifth the ammonia concentration rather than one-half.

In the present case, the difference between the approximate and more precise concentrations is only a factor of two. For some applications this might not be considered serious. For those complexes in which the number of ligands is larger, the difference is correspondingly greater. Even so, the use of the instability constant in this manner represents a serious error in theory and should not be continued.

A further complication arises from the hydrolysis of the ammonia which is liberated by the dissociation of the complex ion. This hydrolysis has been disregarded in both of the above calculations. If it were taken into account, it would further complicate an already laborious task. Since solutions of the pure complex ion are rarely encountered in the laboratory, the computation is seldom necessary.

The dissociation of complex ions contrasts with that of polyprotic acids in one very important regard. The successive ionization constants for polyprotic acids usually differ from one another by a factor of about 10^3 to 10^5. This means that, unless the highest accuracy is required, calculations need to treat only one step of dissociation at a time. On the other hand, the successive constants for complex ions are all of the same order of magnitude. For the diammine silver complex, the second step dissociation constant $(1/k_1)$ is actually larger than that for the first $(1/k_2)$.

Bjerrum's data on the silver-ammonia system have been plotted in Figure 1 to show the mole fraction of each silver-containing species as a function of the free ammonia concentration. The total silver concentration was 0.0200 *M*. The medium was 2 *M* ammonium nitrate. The free ammonia concentration, $[NH_3]$, was changed by adding small amounts of pure ammonia

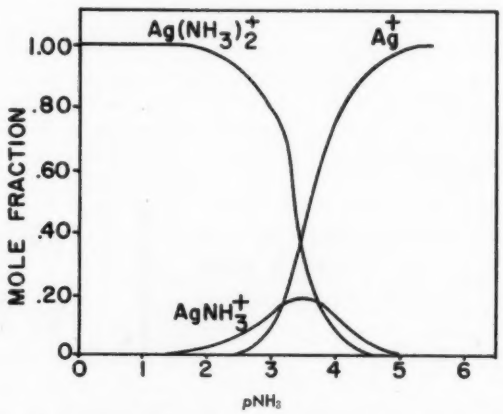

Figure 1. Proportion of the different silver species as a function of pNH_3.

and was measured by means of a glass electrode. Figure 2 is a similar plot for the typical diprotic acid, oxalic acid. The dissociation constants for oxalic acid are $K_1 = 6.5 \times 10^{-2}$ and $K_2 = 6.1 \times 10^{-5}$ (*3*).

In Figure 2 it can be seen that at *p*H values of less than 2 or more than 3, only two species are present in an amount great enough to be taken into account. At values between 2 and 3 a single species, $HC_2O_4^-$, predominates.

On the other hand, Figure 1 shows that as the concentration of ammonia decreases from left to right, the concentration of the diammine decreases while the concentration of the monammine at first increases, then decreases. The concentration of silver ion at first is less than that of monammine, but eventually it becomes the only silver-containing species present in significant concentration. There is no intermediate

range of pNH_3 in which the monammine can be neglected in comparison with either the silver or the diammine. In fact, the only justified neglect can be that of the silver at pNH_3 values below 2.5 or that of the diammine at pNH_3 values above 4.5. It is the existence of appreciable amounts of the monammine in the pNH_3 range between 1.5 and 5.0 which renders calculations for the dissociation equilibria difficult.

Calculations Involving Complex Ions

Despite the difficulties discussed in the foregoing section, it is possible to perform meaningful calculations for the dissociation and formation of complex ions. In the laboratory work associated with beginning chemistry and qualitative analysis courses, complexes are nearly always formed in a relatively large excess of complexing agent. Moreover, the complexes which are useful have reasonably large formation constants; that is, the complexes are relatively stable.

If the above two conditions are met, than the following two assumptions can be made for the purpose of calculation:

(1) Virtually all of the central group (metal ion, ordinarily) is in the complex species with the highest coordination number. The concentrations of free central group and intermediate complexes are very much smaller.

(2) The concentration of free ligand is determined by the excess of complexing agent. The free ligands released by the dissociation of the complexes may be neglected.

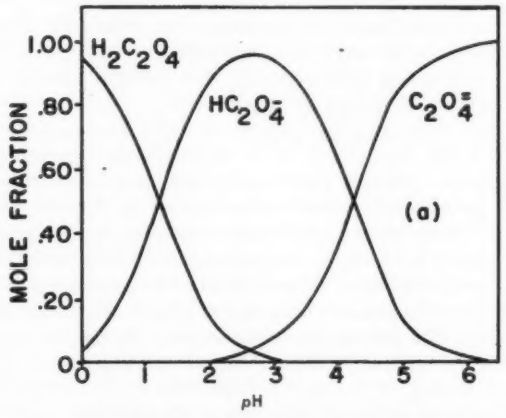

Figure 2. Proportions of the different oxalate species as a function of pH.

Under these conditions the instability constant expression becomes a valid representation of the actual situation and may be used for purposes of calculating the central group concentration.

Consider this problem as an example of the application of these assumptions:

Calculate the concentration of silver ion in a solution prepared by dissolving 0.010 mole of $AgNO_3$ in a liter of buffered solution that is 2 M in both ammonia and ammonium nitrate. $K_{inst} = 9.3 \times 10^{-8}$ at 30° C (2). According to assumption (1)

$$0.010 \simeq [Ag(NH_3)_2^+]$$

and by assumption (2)

$$2 \simeq [NH_3]$$

Substituting into the instability constant expression

$$Ag^+ = \frac{(9.3 \times 10^{-8})(0.010)}{2^2} = 2.3 \times 10^{-10}\ M$$

The assumptions can now be examined to check their validity. For the silver this relationship must hold:

$$0.010 = [Ag^+] + [AgNH_3^+] + [Ag(NH_3)_2^+]$$

From this it follows that

$$[Ag(NH_3)_2^+] = 0.010 - [Ag^+] - [AgNH_3^+]$$

The concentration of free silver ion is obviously negligible in comparison to 0.010 M. From Figure 1 the concentration of the monammine silver ion can be seen to be very much less than 0.1% of the total amount of silver, a concentration which is also negligible. Nearly all of the silver has been completely complexed by the excess ammonia; assumption (1) is justified.

Ammonia in the solution will be either free or in complex ions. The equilibrium

$$NH_3 + H_2O \rightleftharpoons NH_4^+ + OH^-$$

will be shifted to the left by the high concentration of ammonium nitrate. If the entire 0.010 mole of silver ion were in the form of the diammine silver complex ion, only 0.020 mole of ammonia would be required. The remaining concentration is 1.98 M, a value which does not differ significantly from 2. Therefore, assumption (2) is justifiable.

Conclusion

Since the successive formation constants for complex ions are of the same order of magnitude, precise equilibrium calculations are very difficult. Although only the diammine silver complex has been used here for the purpose of illustration, the same argument holds true for other complex ions. Under the conditions ordinarily encountered in the laboratory, simplifying assumptions can usually be made which keep the computations within the ability of the beginning student in chemistry. When the theoretical presentation of complex ion equilibria is made, the discussion should correctly acknowledge the stepwise formation and dissociation of the ions. Problem solving should then be restricted to conditions under which the simplifying assumptions are justified. In this way the need for correct theoretical approach with reasonably simple mathematics can be met.

Acknowledgment

The author wishes to express his gratitude for the helpful suggestions offered by Dr. Karol J. Mysels, the editor of this column.

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Vibrating Molecular Models

Frequency shifts in strained ring double bonds

Carbonyl groups and double bonds in strained rings have been extensively studied in the literature. Their vibrational frequencies in the infrared and Raman spectra are observed to be displaced from the comparable positions in unstrained or noncyclic compounds (*1*, *2*). Calculations on ketones by Halford (*3*) and Bratoz and Besnainou (*4*) indicate that the bond angle changes in rings will shift the C=O frequency due to mechanical effects, Bratoz specifying that most of the shift is due to changes in double bond-single bond mechanical interaction. The frequency shifts have alternately been ascribed to changes in the double bond force constant as a result of strain.

It was decided to study the general effect of double bond-single bond interaction using vibrating molecular models.

Vibrating mechanical molecular models were first used by Kettering, Shults, and Andrews (*5*) to demonstrate and study modes of vibration in various molecules. Basically, the nuclei are represented by weights and the interatomic bonds by helical springs. When this model is freely supported and connected through a loose coupling to an eccentric on a variable speed motor, the model will perform all the normal modes of vibration. When the "disturbing" frequency of the eccentric oscillation matches one of the natural vibrational frequencies of the model, resonance occurs and the model vibrates. At other frequencies, the model remains quiet.

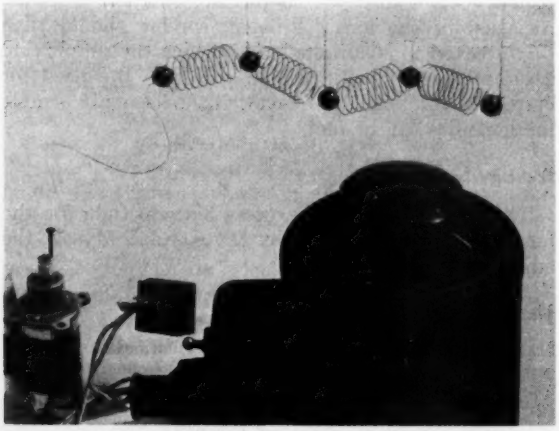

Figure 1. The photograph shows the apparatus for a mechanical analogy of molecular vibrations. The dc motor is connected to a variac through a selenium rectifier. An eccentric in the form of a nail on the side of the motor shaft is connected to the suspended molecular model (the skeleton of *n*-butane in this case) through a coupling wire.

If the models are correctly made and supported, and their limitations realized, they can be studied profitably.

There are many ways to construct models, one of which is as follows. Spring wire 16 mils in diameter was wound into a helical coil about $^{7}/_{8}$ in. in diameter. A single bond included 9 coils at the end of which the wire was shaped to hold a $^{7}/_{16}$-in. diameter steel ball within suitable wire turns, on the spring axis, after which the wire continued for 9 more coils to form the next bond. "Double bonds" consisted of two "single bond" springs. "Atoms" were about $2^{1}/_{2}$ in. apart. These were low frequency models, about 7 cycles per second for the C=C stretching vibration.

An alternate set of high frequency models (about 50 cycles per second) using heavier weights (1 in. diameter) and stiffer springs (0.4-in. wire diameter, $^{7}/_{16}$-in. coil diameter) was also made. The ends of the springs were brought to the axis and extended into holes in the "atoms." "Double bonds" consisted of single springs twice as stiff as the springs used for "single bonds."

The stretching and bending force constants of the springs were measured by suspending a weight from one end of a spring held vertically and horizontally by the other end and noting the strain.

The ratio of stretching to bending force constants was made about 8 to 1 to approximate the average molecule. This is most easily adjusted by stretching the springs out somewhat, increasing all the coil spacings. This does not disturb the relative molecular geometry but lowers the bending force constant relative to the stretching until the desired ratio is obtained.

Since only planar vibrations of planar molecules were studied, the models were suspended with the molecular plane horizontal. Every "atom" was supported by a long thread to a support high enough for pendulum frequencies to be relatively low. Thus, every "atom" is free to move within the plane.

A dc motor with reduction gears was connected through a selenium rectifier to a variac. A nail was taped to the motor shaft to provide an eccentric. One of the double bonded carbon atoms of the model and the eccentric were connected by a large turn of very fine wire (10 mils). As the motor speed varied and resonance was obtained, the normal modes were performed and the frequencies measured with a stroboscope. For the high frequency models, a motor without reduction gears was used.

In this study, the effect of only one variable was investigated, namely the change in double bond-single bond interaction as a function of angle. The weights were all the same, the double bond springs were twice as stiff as single bond springs, and the ratio of stretching to bending force constants was about 8 to 1. Thus, frequency shifts, if any, would be due to mechanical effects rather than to changes in force constant, as

Table 1

Model	Double bond-single bond angle	Description of vibration	Model freq. cm.$^{-1}$	Molecule freq. cm.$^{-1}$	Analogous molecule	Ref.
	180°	$^2/_3$ C=C, $^1/_3$ sym. C—C, out of phase	1835	...	Hypothetical	...
	120°	$^5/_6$ C=C, $^1/_6$ sym. C—C, out of phase	1660	1646	Cyclohexene	*2*
...	...	No model made	...	1611	Cyclopentene	*2*
	90°	C=C (no C—C stretch)	1560	1566	Cyclobutene	*2*
	60°	$^5/_6$ C=C, $^1/_6$ sym. C—C, in phase	1640	1641	Cyclopropene	*13*
	0°	$^1/_2$ C=C, $^1/_2$ C—C, in phase	2100	1974	Acetylene	*9*
	90°	C=C (no C—C stretch)	1575	...	Hypothetical	...
	120°	$^5/_6$ C=C, $^1/_6$ sym. C—C, out of phase	1660	1651	Methylene cyclohexane	*1*
...	...	No model made	...	1657	Methylene cyclopentane	*1*
	135°	$^3/_4$ C=C, $^1/_4$ sym. C—C, out of phase	1700	1678	Methylene cyclobutane	*1, 12*
	150°	$^2/_3$ C=C, $^1/_3$ sym. C—C, out of phase	1740	1781	Methylene cyclopropane	*8, 11*
	180°	$^1/_2$ C=C, $^1/_2$ sym. C—C, out of phase	1815	...	Hypothetical	...
	180°	$^1/_2$ C=C, $^1/_2$ sym. C—C, out of phase	1950	1980	Allene	*10*

force constants were unchanged. Frequencies were measured in cycles per minute and were multiplied by a single proportionality constant (different for the high and low frequency models) to give wavenumbers. The constant was chosen which gave the best wavenumber fit between the models and the molecules. Sample calculation: 410 c.p.m. × 4.05 = 1660 cm^{-1}; 385 c.p.m. × 4.05 = 1559 cm^{-1}.

The results are listed in Table 1. Similar results for both internal and external double bonds were obtained with the two quite different types of high and low frequency models. The models for the unsubstituted internal double bond C—CH=CH—C were made with and without "hydrogen atoms." The hydrogen atoms had little effect on the trends noticed. In the external double bond system the hydrogens with their non-changing small interaction were left out so the system also resembled the ketone carbonyl system in its trend.

As was suspected, in most of the models (allene excepted) the carbon atoms attached only to single bonds did not move much during the double bond stretching vibration; hence, the relatively small mass effects when these masses are changed to masses greater

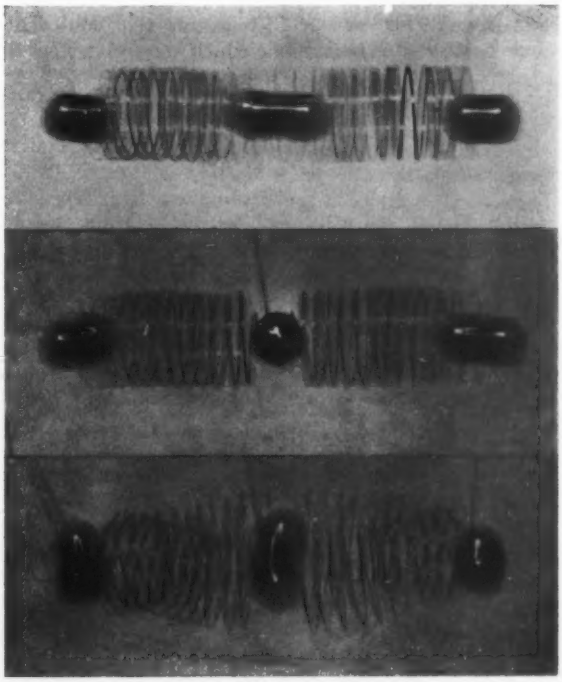

Figure 2. The photograph shows the response of a ball and spring model of CO_2 to varying "disturbing" mechanical frequencies. In the asymmetric stretching vibration (top) and bending vibration (bottom) the two end "atoms" move in the same direction while the middle "atom" moves twice as far and in the opposite direction. In the symmetric stretching vibration (middle) the center atom stays motionless as the end atoms move in opposite directions. The mechanical frequencies are proportional to the actual molecular frequencies. They occur at about 1.4 cycles per second for bending, 2.7 c.p.s. for symmetric stretching, and 4.6 c.p.s. for asymmetric stretching.

than carbon (*4*), and hence the localization of the double bond vibration to the atoms studied. Since the attached atoms do not move much, the attached single bonds must be altered in length as the double bond stretches and contracts.

When the double bond-single bond angle is 90°, there is no stretching interaction as the double bond stretching vibration merely bends the bond angle.

As the double bond-single bond angle becomes larger than 90°, the single bonds must be increasingly contracted as the double bond stretches. This added resistance to motion increases the frequency which reaches a maximum for the 180° bond angle case. This is an out-of-phase interaction of the double bond stretching vibration with the symmetrical single bond stretching vibration.

As the double bond-single bond angle becomes less than 90° the single bonds must be increasingly stretched as the double bond stretches (in-phase interaction). The added resistance to motion again increases the frequency relative to the 90° case.

The cyclic internal double bonds studied were unsubstituted (C—CH=CH—C). In molecules, further changes occur when the hydrogens are substituted. Cyclobutene absorbs at 1566 cm^{-1} (*2*) but various 1,2-dimethyl cyclobutenes absorb at 1685 cm^{-1} (*6*). This is extremely difficult to explain if the cyclobutene double bond is weakened by strain but easy to explain in terms of additional double bond-single bond interaction in the substituted case. The same thing occurs in the cyclopentene case. Cyclopentenes with the two double bond hydrogens intact absorb at 1617–1614 cm^{-1}, 1-alkyl cyclopentenes absorb at 1657–1650 cm^{-1}, and 1,2-dialkyl cyclobutenes absorb at 1686–1671 cm^{-1} (*7*). From the 6 membered ring to the 4 membered ring, the 1,2-dialkyl cyclo-enes absorb in nearly the same place at 1690–1670 cm^{-1}. This is markedly different from the unsubstituted rings. The decrease in the cyclic single bond-double bond angle is somewhat compensated for by an increase in the non-cyclic single bond-double bond angle which keeps the effect of interaction nearly constant. When the cyclic angle gets below 90°, however, the effect of interaction is to increase rather than decrease the frequency, and this combined with a non-cyclic single bond-double bond angle in cyclopropene of 149° 55′ (*14*) will sharply increase the frequency of 1,2-disubstituted cyclopropenes. These are found at 1900–1880 cm^{-1} (*15*, *16*). In the same manner, dimethyl acetylene absorbs at 2313 cm^{-1} (*17*, *18*) compared with 1974 cm^{-1} for acetylene (*9*).

Thus, it seems that the change in double bond-single bond interaction as a function of bond angle is sufficient in itself to explain most of the frequency shifts, and is the most important variable involved.

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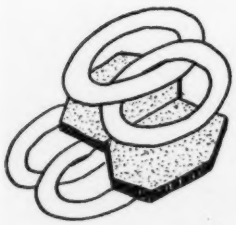
A Chem Gem



Laboratories at Los Angeles Valley College announce discovery of a novel type isomerism existing in the π-bonds of naphthalene. Higher rings are under study now to extend scope of this elegant investigation line.

Namar spectra have shown that when naphthalene is toasted in a bakery oven, a mothball sandwich forms (see figure). Under these conditions, π-electrons above and below the ring system coalesce with nodes bilinear, creating inter- and outer-locked pretzel bonds which give rise to *cis-trans* pretzelism (a new twist on donut isomerism). Adding NaCl to these solutions immediately precipitates the compound since the pretzels are salted out. Delta Eat for the reaction is negative.

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A Simple Demonstration of Some Precipitation and Solubility Effects

Although the solubility product principle for sparingly soluble salts is considered in every general chemistry textbook, the effects due to complex ion formation are frequently neglected. An understanding of these is often quite useful in explaining many phenomena encountered in practical laboratory and industrial situations. For example, in testing wash water for the absence of chloride or silver, it is possible to get a negative test even in the presence of these ions if the ionic concentrations of added Ag^+ or Cl^-, respectively, are too high and fall in the region of complex solubility.

Furthermore, when the solubility product is exceeded and precipitation ensues, there are various processes that can determine the physical nature of the precipitate. In some cases, a coarse precipitate is obtained that settles out rapidly; in others, a colloidal suspension that runs through even a fine filter is produced. If the colloidal particles are exceedingly small, the turbidity in diffuse light may appear so low that the sol can actually be mistaken for a true solution. Certainly, the turbidity alone is no indication of the amount of precipitate since it depends upon the particle size.

We have developed a very simple yet striking experiment which demonstrates a number of these precipitation and complex solubility effects. The purpose of this paper is to describe this experiment and to provide an explanation of the basic physical processes involved. Silver bromide is used in this particular case although silver iodide is equally effective. Silver chloride, however, is not as effective in this demonstration.

The solubility of silver bromide and its precipitation effects have been studied quite extensively and in Figure 1 (upper) we have drawn the complete solubility curve in water at 20°C for various concentration ratios of both precipitating components ($AgNO_3$ and KBr or NaBr) (on a log-log plot).[1] The curve encloses the precipitation range of silver bromide; outside of it silver bromide is not precipitated.

First it should be emphasized that, aside from the well-known limitations due to the influence of the activity coefficient, the simple solubility product principle has a limited utility. Any formation of complex ionic species involving the precipitating ions (Ag^+ and Br^-) means that the concentrations of these ions are no longer given by the concentrations of the precipitating components.

This work has been supported by the Office of Ordnance Research Contract No. DA-ORD-10.

[1] See LINKE, W. F., *in* SEIDELL, A., "Solubilities of Inorganic and Metal-Organic Compounds," 4th ed., Vol. 1, New York, 1958. Data are especially taken from: KRATOHVIL, J., ET AL., *Arhiv kem.*, **26**, 191 (1954); LIESER, K. H., *Z. anorg. u. allg. Chem.*, **292**, 97 (1957).

Consider, for example, the solubility point at equivalent concentrations (Figure 1) indicated by the circle. It is obvious that only for the linear range (perpendicular to the equivalence line) on either side of this point can we use the concentration of the precipitating components in the solubility product expression

$$K_{sp} = [Ag^+] \cdot [Br^-] = 2.67 \times 10^{-13} \qquad (1)$$

We will refer to this as the *normal solubility region*. In this range the equilibrium can be described completely by

$$AgBr \rightleftarrows Ag^+ + Br^- \qquad (2)$$

With greater excess of either precipitating component, the solubility curve deviates sharply from the straight line showing a tremendous increase in the solubility of silver bromide. If the concentrations of the precipitating components are used to calculate the solubility product, the values exceed the solubility product constant by several orders of magnitude and yet optically clear solutions are obtained. This is explained by the formation of soluble complex ionic species of silver bromide, such as $AgBr_2^-$, $AgBr_3^{-2}$, etc. (in excess of bromide ions) and Ag_2Br^+, Ag_3Br^{+2}, etc. (in excess of silver ions). Complex ionic equilibria of similar nature with AgCl were recently described in THIS JOURNAL.[2] Accordingly, the solubility along the curved part of the solubility diagram is designated as *complex solubility*.

Solid silver bromide is precipitated only when the precipitating components are mixed in concentrations within the solubility curve. This area is the site of various precipitation processes, all of which result in the formation of crystalline silver bromide particles. However, their size and surface properties (charge and charge density) will vary considerably. Some of the most important regions are marked in the solubility diagram. Along the equivalence line there is a narrow range, designated as the *equivalent region*, where the precipitation process is very fast, resulting in relatively coarse particles which are either neutral or very weakly charged. This range is also known in analytical chemistry as Gay-Lussac's turbidity maximum and is used in the quantitative analysis of halides or of silver. On both sides of this equivalent range is a rather extensive area in which very small and strongly charged colloidal AgBr particles (primary particles) are formed upon mixing of the precipitation components. These particles are positive in excess of Ag^+ and negative in excess of Br^-. Such colloidal suspensions are stable over a long period of time and exhibit an exceedingly low turbidity. These are designated as the *stability*

[2] RAMETTE, R., J. CHEM. EDUC., **37**, 348 (1960).

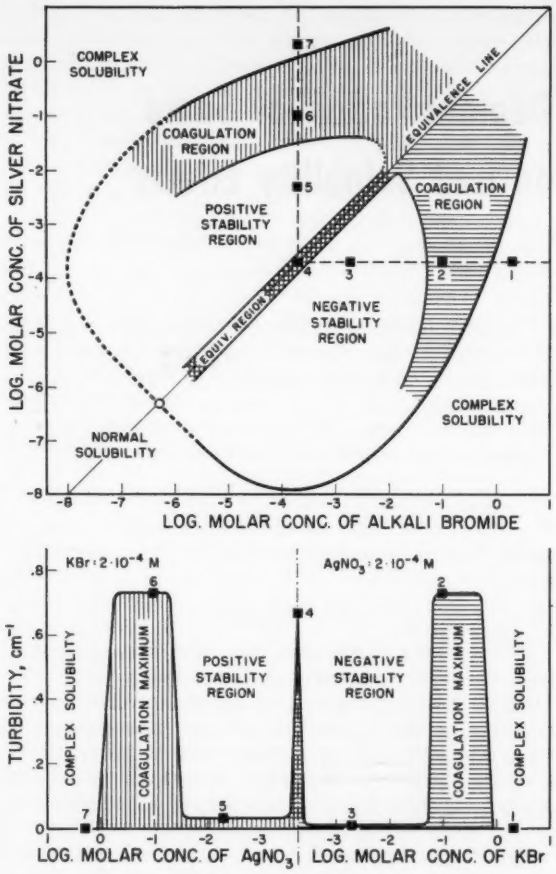

Figure 1. Upper: *Solubility curve* of silver bromide in water at 20°C obtained by mixing silver nitrate with alkali bromide. Full part of the curve has been obtained experimentally using either KBr or NaBr.[1] Both salts give solubility points on the same curve. No data are available for the dotted part of the curve which has been drawn analogously to the silver chloride solubility curve for which complete data are available.

Lower: *Precipitation curve* of silver bromide. Turbidity, measured 10 minutes after mixing of precipitating components using light of wavelength $\lambda = 546$ mμ, is plotted for various concentrations of KBr and $AgNO_3$ corresponding to the dashed line in the upper diagram. In the middle of the diagram both components are present in equivalent concentrations. On the right-hand side silver nitrate is present in constant concentration (2×10^{-4} N) and the concentration of KBr gradually increases. On the left-hand side KBr is constant and $AgNO_3$ increases. Blackened squares in both diagrams represent the concentrations used in the demonstration and described in detail in Table 1.

regions. Finally, at still higher excess of either precipitating component (but lower than that necessary to produce complex solubility), coarser particles are again produced, and the systems are correspondingly highly turbid. These two ranges, indicated by hatching in the precipitation diagram, are usually denoted as the *coagulation regions.* This is because the silver bromide particles are actually aggregates formed by coagulation of the primary particles in the presence of the high electrolyte concentration (potassium ions coagulate negatively charged, and nitrate ions coagulate positively charged AgBr sol particles).

It is quite obvious, then, that mixing potassium bromide with silver nitrate solutions will produce a variety of effects depending upon the concentrations of these solutions. The turbidity, which in turn depends upon particle size, will vary over a wide range, and thus can be used to detect and to demonstrate these effects.

A convenient way of presenting these results is to plot the turbidities of a number of solutions in which

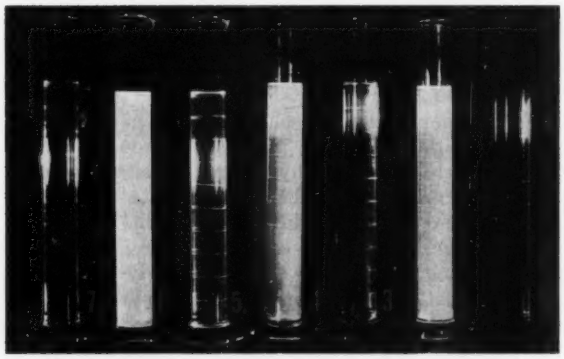

Figure 2. A photograph of cylinders (1 to 7) containing solutions as described in Table 1.

one component is kept constant whereas the concentration of the other component is gradually increased, starting with the equivalent concentration. This has been done, for example, in the *precipitation curve* in the lower diagram of Figure 1. The left part of this curve is for a constant concentration of KBr and the remainder for a constant concentration of $AgNO_3$. The dashed lines on the upper diagram correspond to the concentrations along the abcissa in the lower diagram. Three precipitation maxima and four minima are clearly discernible. They are again described in the legend.

Table 1. Summary of Experiments Performed
(100 ml of $AgNO_3$ solution added to 100 ml KBr solution in each case)

System	1	2	3	4	5	6	7
Initial conc. of solutions:							
$AgNO_3$ (M)	4×10^{-4}	4×10^{-4}	4×10^{-4}	4×10^{-4}	1×10^{-2}	2×10^{-1}	4
KBr (M)	4	2×10^{-1}	4×10^{-3}	4×10^{-4}	4×10^{-4}	4×10^{-4}	4×10^{-4}
Final conc. in sample:							
$AgNO_3$ (M)	2×10^{-4}	2×10^{-4}	2×10^{-4}	2×10^{-4}	5×10^{-3}	1×10^{-1}	2
KBr (M)	2	1×10^{-1}	2×10^{-3}	2×10^{-4}	2×10^{-4}	2×10^{-4}	2×10^{-4}
Observations:	Completely clear	Strongly turbid	Very weakly turbid	Strongly turbid	Very weakly turbid	Strongly turbid	Completely clear
Result of adding $MgSO_4$ soln:			Becomes very turbid		Becomes very turbid		

In order to obtain a complete picture of these precipitation effects of silver bromide a three dimensional model can be used, in which the solubility diagram (Fig. 1, upper) would be the base, and the precipitation curve (Fig. 1, lower) a cross-section. Such a model has been built and presented elsewhere.[3]

Demonstration Experiment

We will now describe an experiment that can be used to demonstrate these effects. It can be performed by simply mixing solutions of KBr and $AgNO_3$ at carefully selected concentrations. A suggested series of seven such mixtures is given in Table 1. The systems correspond to the blackened squares in both diagrams of Figure 1 and are numbered from 1 to 7; the same numbers are used in Table 1. It is obvious that solutions 1 and 7 are in the region of *complex solubility* and despite the high concentration of KBr and $AgNO_3$ they will remain absolutely clear. Systems 2 and 6 are *coagulation regions* and they will be quite turbid; the large AgBr aggregates scatter the light intensely. Systems 3 and 5 are in the regions of stable silver bromide sols (either positively or negatively charged) and will appear quite clear. It should be pointed out to students that systems 3 and 5 contain approximately the same amount of silver bromide precipitate as systems 2 and 6, but since the particles are much smaller, they do not scatter light appreciably. However, even these do exhibit a Tyndall beam under intense illumination. This can be used to distinguish them from the true solutions in tubes 1 and 7. Finally, system 4 appears quite turbid immediately after mixing, being in the range of the equivalent (isoelectric, Gay-Lussac) maximum.

Other volumes than suggested in the table can be used as long as the concentrations are kept unchanged. It is suggested that system 4 (equivalent concentrations) be mixed first and that the mixing then proceed from this in either direction. The concentration of solutions in the equivalent range must be rather accurate because of the narrowness of the isoelectric precipitation range. A small excess of silver nitrate (no more than 3% of concentration of AgBr) will produce strongest turbidity. The fact that the equivalent maximum is slightly shifted toward excess silver ions is well known. However, if bromide ions are in slight excess, the silver bromide particles will become so strongly charged that a stable sol of low turbidity will result.

In Figure 2, cylinders containing the seven systems described above have been photographed in an intense beam of light. If one mixes the middle system first (equivalent concentrations) and then proceeds from this outward in either direction, one gets in sequence (4) high turbidity due to formation of precipitate, (3 or 5) nearly clear solution, due to formation of a very fine, stable colloidal suspension, (2 or 6) high turbidity due to formation of a coarse colloidal suspension, and (1 or 7) clear solution due to complex ion formation. In proceeding from the center outward, one is mixing a fixed concentration of one component with increasing concentration of the second component. Actually, if the instructor asks the students what will happen on the basis of simple solubility product considerations, everything goes against the expectation. However, there is a logical explanation in each case.

An additional experiment can easily be done. A few drops of concentrated solution of a "neutral" (coagulating) electrolyte (such as $MgSO_4$) can be added to systems 3 and 5 (after the mixing of all systems has been completed). In a very short time these nearly clear solutions will become as turbid as systems 2 and 6. This increase in turbidity is caused by coagulation of stable sols (2 and 6) by means of Mg^{+2} and SO_4^{-2} counterions, respectively.

If more than seven systems are chosen, a complete curve, as given in Figure 1 (lower), can be obtained. This can be done quantitatively as a laboratory experiment using a spectrophotometer for turbidity measurements. A convenient wavelength is 546 mμ.

Because the differences in turbidities are great enough, visual observation will suffice for classroom demonstrations. It should be obvious that Figure 1 offers a number of other combinations for demonstrations from which a teacher may choose according to his interests.

[3] MATIJEVIĆ, E., *Chimia (Switz.)*, **9**, 287 (1955); see also *Disc. Faraday Soc.*, **18**, page facing p. 223 (1954).

What Is It?

See page 408.

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A Paper Chromatographic Scheme for the Identification of Metallic Ions

Several schemes of qualitative analysis using paper chromatography have been elaborated already. Pollard, *et al.* (*11, 12, 13*) have employed paper chromatography in combination with spot tests for the detection of the common cations. Whenever several ions do not separate they are distinguished by the use of color reagents on parallel chromatograms. Other authors (*2, 14*) have proposed the classical group separation followed by the separation of each group by paper chromatography.

Recently, in several papers concerning the identification of organic substances, for example phenols by Reio (*15*) and aromatic compounds by Gasparic and Vecera (*4*), the graphical representation of R_f values of an ion in a number of solvents has been used to achieve identification by the characteristic shape of the graph. The term "chromatographic spectra" has been applied to such representations. While he welcomes the principle and is pleased to use it for his purpose, the author considers that the term "spectrum," which has strong associations to phenomena relating to light, is less suitable than the term "profile." It seems unnecessary to further confuse a subject which already is confused by the very term "chromatography" itself. It is proposed to use henceforth in this communication the term "chromatographic profile." The author believes that this concept has not been applied previously to metal ions.

Apart from its use as a general qualitative method, the scheme is considered to be of interest and value in a number of ways. It lends itself to the identification of small amounts of cations in mineralogy and geochemistry, to the identification of small inorganic residues of organic substances, and to the identification of inorganic cations in many biological fields. In these applications, and no doubt in many others, analysis by paper chromatography often seems desirable but, as yet, no adequate directions have been given in the literature to non-chemists who could profit from this kind of analysis. This paper is presented partly to fill this need and partly as a step toward a complete system of qualitative analysis by paper chromatography. Thus it combines some new concepts with what might appear to some to be elementary details.

The author also has tried to counter a common criticism of some modern analytical schemes, namely, that they have no educational value. If fact the reagents and solvents have been so selected as to illustrate several of the modern concepts of the chemistry of complexes. In addition, for the identification of most of the ions, it has been possible to use as reagents only ammonium polysulfide and 8-hydroxyquinoline. Special tests have been kept to a minimum. Of educational value for students, there are the opportunities to distinguish metals forming colored sulfides, colored hydroxyquinolates under a variety of *p*H conditions, fluorescent hydroxyquinolates similarly and non-fluorescent hydroxyquinolates. The effect of the electronic structure of the metal ion upon fluorescence in metal hydroxyquinolates has been discussed recently by Stevens (*17*), who has shown that the hydroxyquinolates that fluoresce most readily are those derived from cations containing the most stable electro grouping (that is, with shells either full or empty). The solvent ethanol-methanol-2*N*.HCl serves to show the behavior of ions in a weakly acid, polar medium allowing hydration to differentiate between such groups of ions as Mg-Ca-Sr-Ba- or Y-La-Sc. Both butanol-HCl-HF and acetone-HCl-HF solvents illustrate the behavior of the insoluble fluorides of the rare earths and of the fluoride complexes of Nb, Ta, Zr, and Hf. Butanol-HBr (together with other solvents) may be used to illustrate the increased tendency to complexation of the B-group elements with increasing atomic weight of the halogen. Acetone-HCl-HF can introduce also the complexing sequence in the transition elements such as the R_f value sequence Ni-Mn-Co-Cu-Fe Zn. It is noteworthy that the behavior of the cations conforms closely to the geochemical classification of the elements by Goldschmidt (*6*) and the later review of that classification by Anderson (*1*). The lithophile metals display generally low R_f values in the three solvents containing concentrated acids and high values in the remaining one which contains dilute acid. This illustrates their limited ability to form complexes. In contrast the chalcophile metals, in keeping with their general ability to form complexes, display generally high R_f values in all solvents.

With only a few exceptions (notably Zr-Hf, La-Y and Sn-Cd) the chromatographic profiles which can be prepared from the data herein are characteristic and permit the identification of the ion. They are to be preferred to color and fluorescence which vary so much with concentration. However, the properties of color and fluorescence revealed by the methods of detection of the ions permit a convenient grouping of the data into an analytical scheme (Table 1, columns 9, 10, 11, 12).

Lastly, because of the small quantities required, the scheme affords an economical means of introducing to students the analytical chemistry of some of the more expensive metals.

The Solution of the Ions

It is assumed that the ions to be tested will be already in solution. However, the following recommendations

are made for the various solvent mixtures:

Chlorides may be used in all solvents.
Excess of nitrates, sulfates, and phosphates is to be avoided.
For solvents 1 and 4, HF may be present in the solution to be analyzed. The use of HF cannot be avoided in the case of Nb and Ta.

Preparation of the Chromatograms

Whatman's No. 1 chromatography paper was used in the preparation of the data contained in this communication.

Normally, four chromatograms are prepared from each of the four solvents. These can be obtained by hanging in each solvent the lower short edge of four paper strips (3 cm × 25 cm) from polythene hooks inserted in the stoppers of the solvent jars. A spot or stripe of the solution to be analyzed is first placed between two small pencil marks about 1.5 cm from the lower edge of the strip. Any other data necessary should be recorded on the strip in pencil. As an alternative to the strips, a number of spots or stripes may be placed near the base of a rectangle of paper (30 cm × 25 cm) which can be clipped to form a cylinder which can stand in the shallow solvent. The development is carried out by allowing the solvent to ascend the paper to a convenient height. After development the strips (obtained directly or cut from the rectangle) are submitted to reagents to identify the positions taken by the ions.

Solvents

The following solvents as single-phase mixtures were used:

Solvent 1. [Grand-Clement, *et al.* (*5*).]

Butanol	100 ml
HCl conc.	50 ml
HF conc.	2 ml
Water	48 ml

This solvent should be prepared and used in polythene vessels.

Solvent 2. [See Lederer and Lederer, p. 491 (*9*).]

(*a*) The butanol fraction of

Butanol	100 ml
HBr conc.	10 ml
Water	90 ml

(*b*) To the above fraction add conc. HBr 40 ml.

Solvent 3. [Götte and Pätze (*7*, *8*).]

Ethanol	45 ml
Methanol	45 ml
2*N*·HCl	60 ml

Many of the R_f values for this solvent have not been recorded previously.

Solvent 4. [Grand-Clement, *et al.* (*5*).]

Acetone	90 ml
HCl conc.	5 ml
HF conc.	1 ml
Water	4 ml

Solvent 4 should be prepared and used in polythene vessels. The container should be covered to protect it from air currents and temperature changes. The acetone front of this solvent is often difficult to mark on the chromatograms owing to the rapid evaporation of the solvent. After the method of Somfalvy and Ausch (*16*), it was found that if three or four spots of a solution of rosolic acid in ethanol (0.1 mg/ml) are placed on the paper with or near the test solutions, the dye will be carried up at the top of the acetone front and hence the latter can be readily marked at any convenient time. The author has noted that ball-point pen ink serves almost as well as rosolic acid in this regard.

Time of Development

The following development times gave chromatograms of sufficient length to permit reliable determinations of the R_f values (viz., lengths greater than 10 cm).

Solvent 1	4 or 5 hours
Solvent 2	5 hours
Solvent 3	3 to 4 hours
Solvent 4	1 hour

Detection of Ions

General. For a complete analysis the four chromatograms from each solvent are tested as follows:

Chromatogram 1. $(NH_4)_2S_x$ test; $SnCl_2$ test; $SnCl_2$ + KCNS + HCl test.
Chromatogram 2. 8-hydroxyquinoline (*p*H 2–6) test.
Chromatogram 3. 8-hydroxyquinoline (*p*H 6–10) test + sodium rhodizonate test.
Chromatogram 4. Chlorine water test; *p*-dipicrylamine test.

Colored spots on chromatograms. (Table 1, column 1.) Many ions gave colored spots on the chromatograms which can be marked at the moment they are taken out of the solvent. This test is very useful but, since it depends so much on the concentration of the ion, it is not to be relied upon. Spots of different reflectance might be observed sometimes while the chromatogram is still in the jar, but these fade very quickly when taken out. However, the relative positions of the points of application of the solution and the spot can be marked in chinagraph pencil on the jar and transferred later to the chromatogram.

Detection by ammonium polysulfide solution. (Table 1, column 2.) One chromatogram from each solvent is held over a concentrated solution of $(NH_4)_2S$ or dipped into a dilute aqueous solution [Lederer and Lederer, p. 481 (*9*)]. Many ions can be positively detected in this way. If the chromatograms are dipped they should then be placed horizontally on absorbent paper to prevent displacement of the spots.

Detection by 8-hydroxyquinoline solutions. (Table 1, columns 3 and 4.) The formation of colored or fluorescent insoluble hydroxyquinolates is well summarized by Welcher (*18*), p. 265 *et seq.* and Stevens (*17*). For the purpose of this scheme, two hydroxyquinoline solutions are used and a technique of *p*H variation (while the chromatograms are under ultra-violet light) is introduced.

The first solution is prepared as follows: about 2 g of 8-hydroxyquinoline is partly dissolved in 20 ml of ethanol, leaving an excess of solute. A few drops of sodium acetate solution are added, after which glacial acetic acid is added slowly until the solution becomes clear and yellow. At this stage the *p*H is usually 5 but it may range from 2 to 6. This solution is only effective for about twenty minutes.

The second solution is prepared by partly dissolving the 8-hydroxyquinoline in ethanol as in the first solution. A few drops of neutral sodium tartrate solution are added and then concentrated ammonium hydroxide is added until the solution becomes orange-yellow when the *p*H is usually 10 (although it may range from 6 to 10). The solution only lasts about twenty minutes.

Separate chromatograms are dipped into the solutions and are then placed horizontally on absorbent paper. Two things are worth noting here. First, if the hydroxyquinolate is somewhat soluble (for example Rb, Li, and K), the fluorescent matter might well pass through to the absorbent paper. If the position of the chromatogram is marked on the latter, the position of the spot may be defined from the absorbent paper. For this reason it is sometimes better to spray on the hydroxyquinoline solutions. Second, the hydroxyquinoline solution sometimes forms fluorescent compounds with the metal ions contained in the absorbent paper. These may penetrate to the chromatogram but will become recognizable with experience.

Most of the ions produce either fluorescent or colored hydroxyquinolates. After the colored spots have been marked, the dipped chromatograms are viewed under ultra-violet light (which, of course, must be shielded from the human eye) for observation of the fluorescence. Stevens (*17*) has discussed the fluorescent hydroxyquinolates and has prepared a list of the elements concerned in order of intensity of fluorescence.

It has been found that sometimes the fluorescence of a hydroxyquinolate is intensified or diminished by spraying the chromatogram with concentrated ammonium hydroxide. This treatment, which can be

Table 1. Scheme of Identification and Classification of Cations

Identification								Classification			
Color of spot on chromatogram[a]	$(NH_4)_2S_x$ Color of Spot	8-Hydroxyquinoline Color of Spot	8-Hydroxyquinoline Fluorescence of Spot	R_f Values Solvent 1	Solvent 2	Solvent 3	Solvent 4	Sulfides	Group	Hydroxyquinolates	Ion
1	2	3	4	5	6	7	8	9	10	11	12
	bl	y	fy +	0.77 + c	1.0 + c	0.13 + c	0.59 + c				Ag^{+}
	or to bl-br	y	fy	0.44 + c	0.64 + c	0.45 + c	0.48 + c			Fluorescent	Pb^{+2}
3y 4y	y	g-y	ff y	0.92	1.0	0.96	0.77 to 0.83	Colored	1A	and	Cd^{+2}
3y 4r	br	y	f y	0.75	0.85	0.88	0.85			faintly	Bi^{+3}
	br-bl	y	f y −	0.93	0.95 to 1.0	0.93	0.82			colored	Sn^{+2}
	bl	y	f g	1.0	1.0	1.0	1.0				Hg^{+2}
	y[b]	y - br		0.88	1.0	0.80 + c	0.82				Sb^{+3}
	bl	y to bl		0.92	0.92	0.92	0.65				As^{+3}
1234 y	bl	bl		0.79 to 0.93	0.65 to 0.75	0.70 to 0.73	0.86				Fe^{+3}
13b.g 2p 4g	dk-br	y - br		0.53	1.0 and 0.54	0.65	0.56				Cu^{+2}
123pi 4pi or b	br-bl	br - y		0.45	0.30 to 0.38	0.70	0.45 to 0.55	Colored	1B	Colored	Co^{+2}
24b	y	y		0.73	0.53 + c	0.60	0.83 to 0.92 + c				MoO_4^{-2}
	dk br	y - br		0.41 + c	1.0 + c	0.41 + c	1.0 + c				Tl^{+}
2br	dk br	y		1.0	1.0 + c	1.0 + c	1.0				Tl^{+3}
14g 23y	br-bl	g - br		0.52	0.32	0.63 to 0.67	0.03				Ni^{+2}
123 br	br - bl	br - bl		0.48	0.32	0.70	0.25				Mn^{+2}
		y	ff y-g	0	0.25	0.75	0				Sc^{+3}
		y	ff y	0	0.18 to 0.18 to	0.70 to 0.24	0				Y^{+3}
		dk y	ff g-y +	0 to 0.10 + c	0.19	0.65	0.24 + c				La^{+3}
		y	ff y +	1.0	0.65 to 0.70	0.80	0.63 to 0.68				Ga^{+3}
		y	ff go-y +	0.57	0.91 to 1.0	0.78	0.85 to 0.90				In^{+3}
		y	f y +	0.52 to 0.60 +c	0.12 + c	0.52 + c	0.48 + c	Colorless	2A	Fluorescent and some	Zr^{+4}
		y[c]	f y[e] +	0.59	0.81	0.47	0.29			faintly	Ge^{+4}
		y	f y +	0.40 + c	0.19	0.44	0.02			colored	Ca^{+2}

[a] After solvent development only, numerals refer to solvent number; letters refer to colors. [b] Mostly soluble in alkaline solutions; easily detected otherwise. [c] Faint. [d] Delayed. [e] Fades.

ABBREVIATIONS

y = yellow, or = orange, r = red, br = brown, p = purple, bl = black, pi = pink, f = faint fluorescence, ff = strong fluorescence, b = blue, go = golden, dk = dark, g = green, + = effect augmented by NH_3 spray, − = effect diminished by NH_3 spray, +c = comet

carried out under the ultra-violet light, apparently varies the *p*H; the fluorescence is increased when the optimum *p*H conditions are approached and vice-versa. This diagnostic property has been exploited partly in the tables. The author found this technique very suitable for detecting such ions as Mg^{+2}, La^{+3}, Li^{+}, Ca^{+2}, Sr^{+2} and Zr^{+4}.

Detection by Special Tests. Ions not readily detected by the above methods may be detected as follows:

SeO_3^{-2}, TeO_3^{-2} and TeO_4^{-}:

The chromatogram from the $(NH_4)_2S$ test is dried thoroughly (to drive off the H_2S) and dipped in a solution of $SnCl_2$ in $5N \cdot HCl$. The following spots are diagnostic:

orange	SeO_3^{-2}
dark brown to black	TeO_3^{-2}
brown	TeO_4^{-}
yellow-brown	Pt^{+4}
purple to black	Au^{+3}

Ba^{+2} and Sr^{+2}:

Using a chromatogram from the hydroxyquinoline test, a fresh solution of sodium rhodizonate (0.5 mg in 2 ml water) is sprayed on. Ba^{+2} gives a reddish purple spot and Sr^{+2} a brick-red.

ReO_4^{-}:

The chromatogram from the test above is dipped in a solution of $SnCl_2$ + KCNS + $5N \cdot HCl$. The following diagnostic spots result:

yellow	SeO_3^{-2}
dark brown-black	TeO_3^{-2}
brown	TeO_4^{-}
brown-black	Au^{+3}
yellow	ReO_4^{-}
golden yellow	Pt^{+4}

Ir^{+4}:

To detect iridium, the fourth chromatogram is sprayed with chlorine water (in a fume cupboard). A faint brownish-yellow spot indicates Ir^{+4}.

TeO_3^{-2} and TeO_4^{-}:

The distinction between TeO_3^{-2} and TeO_4^{-} provides a nice example of progressive reduction of the latter by the chromatography paper; the comet thus produced when TeO_4^{-} is present distinguishes it from TeO_3^{-2} which merely produces a spot.

Rh^{+3}:

The chromatogram is sprayed with a mixture of $SnCl_2$-KI in HCl and heated under an infrared lamp. A brown spot indicates Rh^{+3}.

Table 1. Scheme of Identification and Classification of Cations (continued)

Identification								Classification			
Color of spot on chromatogram[a]	$(NH_4)_2S_x$	8-Hydroxyquinoline		R_f Values							
	Color of Spot	Color of Spot	Fluorescence of Spot	Solvent 1	Solvent 2	Solvent 3	Solvent 4	Sulfides	Group	Hydroxyquinolates	Ion
1	2	3	4	5	6	7	8	9	10	11	12
			f y +	0.30	0.13	0.40	0				Sr^{+2}
			f g-y	0.22	0.06	0.24	0				Ba^{+2}
		g-y	ff g-y	0.94	1.0	0.94	0.90				Zn^{+2}
			ff y-g +	0.42 to 0.46	0.36	0.75	0.02				Al^{+3}
			fb +	0.46	0.40	0.75	0.13				Li^{+}
			fy −	0.43	0.23	0.35	0.05				Rb^{+}
			fy	0.46	0.26	0.34	0.03				Cs^{+}
			fb	0.41	0.10	0.46	0.05				K^{+}
		y	fy +	0.88	0.26	0.86	0.82 to 0.94				Nb^{+5}
			fb −	1.0	0.90 + c	1.0 + c	1.0				Ta^{+5}
		y	ff y	0.54 to 0.60	0.60	0.86	0.22				Be^{+2}
		y	ff y +	0.47	0.37	1.0	0.03				Mg^{+2}
		y	fy +	0.53 + c	0.11 + c	0.50 + c	0.46				Hf^{+4}
		y	f or +	0	0.15	0.60	0				Th^{+4}
1234 y		bl		1.0	0.86 to 1.0	0.91	0.97				Au^{+3}
4 br		dk br		1.0 + c	1.0 + c	0.90 + c	0.93 + c				Pt^{+4}
234 br		y[d]		0.83	1.0	0.91	0.90				Pd^{+2}
123 br, 4 or		bl		0.30 + c	1.0 + c	0.70 + c	0.82 + c	Colorless	2B	Colored	Ru^{+}
1234 g		dk g +		0.38	0.23	0.73	0.06				Cr^{+3}
124g 3y		y		1.0 + c	0.30 + c	0.86 + c	1.0 + c				CrO_4^{-2}
		br		0	0.15	0.54	0				Ce^{+3}
3 y br		y-br −		0.64	0.43	0.76	0.64				Ti^{+4}
		br		0.46	0.37 to 0.43	0.76	0.58 to 0.64				UO_2^{+2}
124g 3y-br		br +		0.47	0.43	0.74	0.64				V^{+5}
		br	fy[c]	0.70	0	1.0 + c	1.0 + c				WO_4^{-2}
1234 br				0.47	0.17	0.82 to 0.86	0.03				Ir^{+4}
				0.86	1.0	0.62	0.71 and 0.15			Colorless	TeO_3^{-2}
				0.68 + c	1.0 + c	0.62 + c	0.71 and 0.15 +c	Colorless	3	and non-	TeO_4^{-}
				0.72	1.0	0.72	1.0			fluorescent	SeO_3^{-2}
				0.82	1.0	0.87	0.89				ReO_4^{-}
1234 br				0.64	0.43	0.65	0.05				Rh^{+3}

K^+, Rb^+ and Cs^+:

The following procedure has been found by the author to produce a positive identification of K^+, Rb^+, and Cs^+ on chromatograms. The chromatogram from the previous test is dried thoroughly to remove the chlorine. The acid character of the chromatogram is neutralized by holding it over concentrated NH_4OH solution. The paper is then sprayed with a solution of *p*-dipicrylamine prepared as follows (*10*): Dissolve 0.2 g dipicrylamine in 20 ml of boiling $0.1N \cdot Na_2CO_3$ solution; cool and filter; the reagent lasts for at least a week. The paper is resprayed after five minutes; after a further three minutes it is placed on a glass plate and washed with $0.1N \cdot HCl$ solution. A reddish spot indicates one of the three ions. Faint color distinctions may be discerned with experience, K^+ giving a pinkish-red, Rb^+ a bluish-red, and Cs^+ a yellowish-red spot.

Precautions. When several metal ions are present in the one solution, chromatograms from the four solvents should be examined at the same time so that the matching characteristics of the ions (or their spots) may be noted. Such characteristics include:

color of the spot as the chromatogram is lifted out of the solvent
time for the test reaction to proceed
color changes as the reactions proceed
slight difference in the color of the spots
degree and hue of the fluorescence
effect of ammonia spray on the fluorescence

Table 2 will be found to be of assistance in that ions of approximately the same R_f values in any one solvent can be seen at a glance. The R_f value in the next solvent usually eliminates all but one or two of the possibilities of the first solvent and so on.

Short-cut Methods

When the presence (or absence) of one or more ions in a solution is required to be demonstrated, short-cut methods can be adopted. One or two chromatograms from solvent 4 (because of its rapid development) are often all that are necessary. Alongside the chromatograms of the test solution one can run also the chromatograms of the ion sought. This affords a ready comparison. Each of the solvents has the advantage of separating certain ions from one another. No doubt other short-cut methods will emerge to serve the needs of individual investigators.

Calculation of R_f Values

The R_f values quoted have been obtained by calculations of the ratio

$$\frac{\text{distance traveled by the ion}}{\text{distance traveled by the solvent front}}$$

The distance in the numerator is measured from the point of application of the solution to the center of the spot. When a comet is encountered the R_f to the top of the comet is quoted.

Table 2. Numerical Order of R_f Values for Cations

Solvent 1	Solvent 2	Solvent 3	Solvent 4
1.00 Ga^{+3} Ta^{+5}	1.00 In^{+3} Zn^{+2}	1.00 Mg^{+2} Ta^{+5}	1.00 Ta^{+5} Hg^{+2}
1.00 Au^{+3} Pt^{+4}	1.00 Au^{+3} Pt^{+4}	1.00 WO^{-2}	1.00 CrO_4^{-2}
1.00 CrO_4^{-2} Hg^{+2}	1.00 Pd^{+2} Ru^{+}	1.00 Hg^{+2} Tl^{+3}	1.00 WO_4^{-2}
1.00 Tl^{+3}	1.00 TeO_3^{-2} ReO_4^{-}	0.96 Cd^{+2}	1.00 SeO_3^{-2}
0.94 Zn^{+2}	1.00 SeO_3^{-2} TeO_4^{-}	0.94 Zn^{+2}	1.00 Tl^{+} Tl^{+3}
0.92 Cd^{+2} As^{+3}	1.00 Ag^{+} Cd^{+2}	0.93 Sn^{+2}	0.97 Au^{+3}
0.91 Sn^{+2}	1.00 Sn^{+2} Hg^{+2}	0.92 As^{+3}	0.93 Pt^{+4}
0.88 Nb^{+5} Sb^{+3}	1.00 Sb^{+3} Cu^{+2}	0.91 Au^{+3} Pd^{+2}	0.90 Zn^{+2} Pd^{+2}
0.86 TeO_3^{-2}	1.00 Tl^{+} Tl^{+3}	0.90 Pt^{+4}	0.89 ReO_4^{-}
0.83 Pd^{+2}	0.92 As^{+3}	0.88 Bi^{+3}	0.86 Sb^{+3} Fe^{+3}
0.82 ReO_4^{-}	0.90 Ta^{+5}	0.87 ReO_4^{-}	0.85 In^{+3} Bi^{+3}
0.79 Fe^{+3}	0.85 Bi^{+3}	0.86 Nb^{+5} Be^{+2}	0.83 MoO_4^{-2}
0.77 Ag^{+}	0.81 Ge^{+4}	0.86 CrO_4^{-2}	0.82 Nb^{+5} Ru^{+}
0.75 Bi^{+3}	0.65 Ga^{+3} Fe^{+3}	0.82 Ir^{+4}	0.82 Sn^{+2}
0.73 MoO_4^{-2}	0.64 Pb^{+2}	0.80 Ga^{+3} Sb^{+3}	0.77 Cd^{+2}
0.72 SeO_3^{-2}	0.60 Be^{+2}	0.78 In^{+3}	0.68 Ga^{+3}
0.70 WO_4^{-2}	0.54 Cu^{+2}	0.76 Ti^{+4} UO_2^{+2}	0.65 As^{+3}
0.68 TeO_4^{-}	0.53 MoO_4^{-2}	0.75 Sc^{+3} Al^{+3}	0.64 Ti^{+4} V^{+5}
0.64 Ti^{+4} Rh^{+3}	0.43 Ti^{+4} UO_2^{+2}	0.75 Li^{+}	0.64 UO_2^{+2}
0.60 Be^{+2}	0.43 V^{+5} Rh^{+3}	0.74 V^{+5}	0.59 Ag^{+}
0.59 Ge^{+4}	0.40 Li^{+}	0.73 Cr^{+3} Fe^{+3}	0.56 Cu^{+2}
0.57 In^{+3}	0.38 Co^{+2}	0.72 SeO_3^{-2}	0.48 Zr^{+4}
0.53 Hf^{+4} Cu^{+2}	0.37 Mg^{+2}	0.71 TeO_3^{-2}	0.48 Pb^{+2}
0.52 Zr^{+4} Ni^{+2}	0.36 Al^{+3}	0.70 Y^{+3} Ru^{+}	0.46 Hf^{+4}
0.48 Mn^{+2}	0.32 Ni^{+2} Mn^{+2}	0.70 Co^{+2} Mn^{+2}	0.45 Co^{+2}
0.47 Mg^{+2} V^{+5} Ir^{+4}	0.30 CrO_4^{-2}	0.65 La^{+3} Cu^{+2} Rh^{+3}	0.29 Ge^{+4}
0.46 Li^{+} Cs^{+} UO_2^{+2}	0.26 Nb^{+5} Cs^{+}	0.63 Ni^{+2}	0.25 Mn^{+2}
0.45 Co^{+2}	0.25 Sc^{+3}	0.60 Th^{+4} MoO_4^{-2}	0.24 La^{+3}
0.44 Pb^{+2}	0.24 Y^{+3}	0.54 Ce^{+3}	0.22 Be^{+2}
0.43 Rb^{+}	0.23 Rb^{+} Cr^{+3}	0.52 Zr^{+4}	0.15 TeO_3^{-2}
0.42 Al^{+3} Tl^{+}	0.19 La^{+3} Ca^{+2}	0.50 Hf^{+4}	0.13 Li^{+}
0.41 K^{+}	0.17 Ir^{+4}	0.47 Ge^{+4}	0.06 Cr^{+3}
0.40 Ca^{+2}	0.15 Th^{+4} Ce^{+3}	0.46 K^{+} TeO_4^{-}	0.05 Rb^{+} K^{+} Rh^{+3}
0.38 Cr^{+3}	0.13 Sr^{+2}	0.45 Pb^{+2}	0.03 Cs^{+} Mg^{+2}
0.30 Sr^{+2} Ru^{+}	0.12 Zr^{+4}	0.44 Ca^{+2}	0.03 Ir^{+4} Ni^{+2}
0.22 Ba^{+2}	0.11 Hf^{+4}	0.42 Tl^{+}	0.02 Ca^{+2} Al^{+3}
0.01 La^{+3}	0.10 K^{+}	0.40 Sr^{+2}	0.00 Sc^{+3} Y^{+3}
0.00 Sc^{+3} Y^{+3}	0.06 Ba^{+2}	0.35 Rb^{+}	0.00 Sr^{+2} Ba^{+2}
0.00 Th^{+4} Ce^{+3}	0.00 WO_4^{-2}	0.34 Cs^{+}	0.00 Th^{+4} Ce^{+3}
		0.24 Ba^{+2}	0.00 TeO_4^{-}
		0.13 Ag^{+}	

A few R_f values quoted vary from those published previously. This is due partly to the taking of some R_f values to the top of comets and partly to the author's avoiding sulfates, phosphates, etc., in the sample solutions.

Preparation of Chromatographic Profiles

The chromatographic profile for each ion is obtained by plotting and joining the R_f values of the ion in the solvents on four adjacent columns. The data for this is readily available in Table 1 (columns 5, 6, 7, and 8). Comets may be represented in the normal way ("+ c").

For each ion, in addition to the chromatographic profile, data on its sulfide, hydroxyquinolate, and fluorescence can be shown near the profile. Thus confirmatory data is available readily for each ion.

Classification

The ions are grouped (Table 1, columns 9, 10, 11, and 12) first into colored (Group 1A and 1B) and colorless sulfides (Group 2A and 2B) and second into fluorescent (Group 1A and 2A) and colored (Groups 1B and 2B) and colorless non-fluorescent hydroxyquinolates (Group 3). If chromatographic profiles are prepared and arranged in the same grouping, the best use of the scheme is achieved.

Typical Results

The possibilities of this method of analysis were investigated with the aid of other workers and a large number of trials were made. Beginning with simple salts, the trials were extended to artificial mixtures of increasing complexity and finally to a few minerals. The two test results shown below are typical of the many carried out.

(I) The sample was a white powder, soluble in 5*N* HCl yielding a yellow solution; S. colorless; H. yellow; F. f. yellow (increased by NH_4OH spray); R_f values in solvents in order; 0.53, 0.10, 0.50 + c, 0.51 + c. The profile (Fig. 1) is characteristic for Zr^{+4} or Hf^{+4}. Conclusion: Zr^{+4} or Hf^{+4} (in fact the sample was zirconium nitrate).

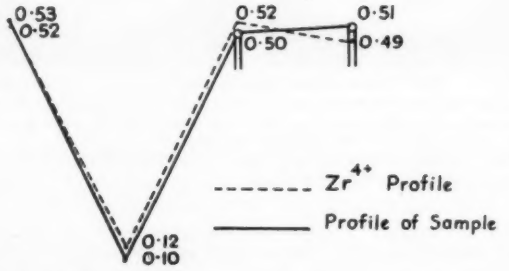

Figure 1. Profile of Sample I.

(II) The sample was a yellow powder, soluble in warm 5*N* HCl. S. brown-black; H. yellow; F. nil; R_f values 0.74, 0.90, 0.89, and 0.85. The profile is close to that of Bi^{+3} (Fig. 2) and on the basis of color of the sulfide and the fluorescence respectively it was possible to reject Cd^{+2} and Sn^{+2} which have similar profiles. Conclusion: Bi^{+3} (which was correct).

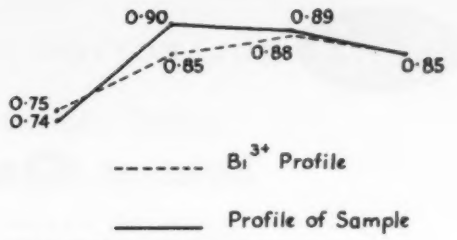

Figure 2. The profile of Sample II.

(III) The sample was a brown solution. There were four sulfide spots with corresponding hydroxyquinolates. Hence:

A.		B.	
S.	dark brown	S.	dark brown
H.	dark yellow	H.	yellow
F.	f yellow	F.	f orange-yellow
R_f	0.94, 0.96, 0.91, 0.67.	R_f	0.74, 0.84, 0.86, 0.80 to 1 + c

C.		D.	
Faint pink out of solvent			
S.	black	S.	brown-black
H.	brown	H.	yellow-brown
F.	—	F.	—
R_f	0.50, 0.38, 0.78, 0.47.	R_f	0.41, 0.32, 0.71, 0.30.

For economy of space the profiles are not illustrated.

Conclusions: A. Sn^{+2} (in spite of low R_f in solvent 4); B. Bi^{+3}; C. Co^{+2}; D. Mn^{+2}.

Acknowledgments

The author wishes to acknowledge with gratitude the assistance given by Dr. M. Lederer by advice, discussion and critical reading of the manuscript. Thanks are also tendered to Signore Misiti and especially to Signorina A. M. Pirelli who carried out most of the tests. The author thanks sincerely the Government of France for the financial assistance given him and for the privilege of working at the Institut du Radium, Paris.

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A Chem Ed COLLECTION

Notes on Qualitative Analysis

EDITOR'S NOTE: The following papers have been collected over a period of months. A similar collection has appeared in previous August issues (J. CHEM. EDUC., **35**, 401 (1958), **36**, 379 (1959), and **37**, 407 (1960)). It is the Editor's hope that collection into one place will prove more attractive to readers than the more frequently used editorial procedure of scattering items such as these through the pages of several issues. We apologize to authors who thus have had to wait to see their work in print.

We commend to readers' attention the fact that often these represent the solution of problems given to freshman students in qualitative analysis courses. We hope that publication in these pages can stimulate just such activity. If readers merely incorporate these ideas into their own laboratory instructions, our battle is only half won. If, beyond that, these ideas suggest to the readers that they use their own ingenuity in similar fashion, we are gratified.

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J. P. Phillips[1]
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Ion Exchange Paper Chromatography

Although experiments in paper chromatography and ion exchange have become fairly common operations in chemistry courses from the high school level up, each method offers certain nuisances for the laboratory instructor. The most serious procedural problem is that both techniques are likely to require more time than the usual laboratory period is designed to cope with.

While circular disc chromatography on paper is faster than strip or sheet methods, the resolution suffers. Rapid flow rates on ion exchange columns cannot be recommended. The preparation and preservation of the solvent mixtures required for paper chromatography is another difficulty, albeit one more likely to trouble the laboratory assistant than the student. Technique and developing equipment also affect the results of conventional one- or two-dimensional sheet chromatography. It is even necessary in many instances to consider the hazards involved in handling some of the required solvents, e.g., their toxicity or inflammability.

Paper chromatography on the rather recently available ion exchange resin impregnated papers[2] is notably more rapid than the usual variety because the resolution of such systems is sufficiently superior to allow good separation, even on circular disc chromatograms (see Figures 1 and 2). Furthermore, only aqueous buffers are necessary for the development, and control of separation is often simply a matter of varying the *p*H of the buffer. Figure 1 illustrates these points well; a mixture of arginine, tyrosine, leucine, and aspartic acids gave acceptable separations on a disc chromatogram requiring only 15–20 minutes to develop. It is very easy for a student to separate two or three amino acids with different isoelectric points. Figure 2 shows the same amino acid mixture developed on ordinary filter paper. While such chromatography performed with aqueous buffers instead of solvent combinations could hardly be expected to be anything but disappointing, the comparison remains striking. Standard disc paper chromatography in Petri dishes gave these results.[3]

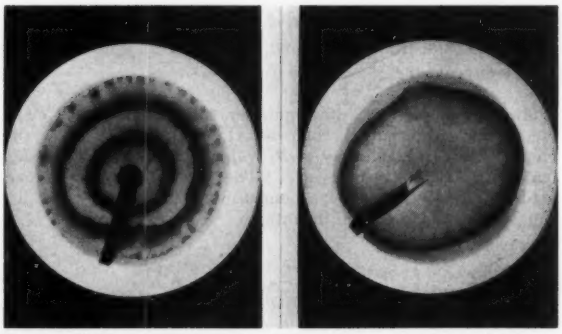

Figure 1 (left). Separation of arginine, tyrosine, leucine and aspartic acid (in that order from the center) with a buffer of *p*H 3.6 on SA-2 cation exchange resin impregnated paper. A ninhydrin dip was used to detect the amino acids; 10 γ of each acid was used.
Figure 2 (right). The same as Figure 1 except that Whatman #4 filter paper was used instead of the SA-2.

A lowering of *p*H in the aqueous buffers used causes a greater tendency of a given amino acid to form a cation, and thus strongly influences its migration on a cation exchange resin impregnated paper. Thus, performance of the experiment at several *p*H values can give widely differing separations.

This experiment is also an effective lecture demonstration.

[1] Present address: Brown-Forman Distillers Corp., Louisville 10, Ky.

[2] The grade used in this work was Reeve Angel SA-2, impregnated with Amberlite 1R-120 resin in the Na^+ form. A bulletin (#3031) is available describing these papers and others, with a bibliography on their use.

[3] For a convenient description of disc chromatography practice, see HART, H., J. CHEM. EDUC., **36**, A501 (1959).

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Preparative Separations Replace Typical Qualitative Analysis

Until a few decades ago, only *inorganic* reagents were used in inorganic qualitative analysis. It was carried out by systematic separations which were essentially a continuous repetition of certain operations, such as precipitation, decantation, filtration, washing, and dissolving. The occupation of the student with qualitative analytical problems was the recommended way of getting a freshman acquainted with the laboratory techniques and with inorganic chemistry.

The Changed Situation

Meanwhile, methods in applied qualitative analysis have changed completely. Nowadays, selective *organic* reagents and novel methods are widely used, e.g., spot tests, paper chromatography, and ion exchange techniques. This causes some pedagogic difficulties: One can teach qualitative analysis in the form which is used today, but then it is necessary to postpone it to a later training period. The alternative is to keep the classical form, but to neglect bringing the student up to date in this branch of chemistry. As a third possibility, the analytical introduction to chemistry can be replaced by inorganic preparation techniques or physicochemical measurements. But in doing so, one loses the chance of putting before each student a different problem. By varying the composition of the samples the instructor charges the student in a certain sense with independent research work.

A New Possibility

However, there is still a possibility of exploiting the pedagogic advantages of the old inorganic analytical training without giving the student a completely wrong picture of today's standard of analytical chemistry. A few years ago, the author[1] suggested replacing qualitative analysis of cations by so-called *preparative separations* in the beginner's laboratory course. The students get a mixture of metal salts, but they do not have to identify the cations which are present. Instead, they are required to deposit the metals as inorganic precipitates in as pure a form as possible. Thus, they are trained in a method which has become most important today for pre-separations in trace-analysis, for separation of radioactive elements, and for preparative work. In the present time, these techniques are more significant than the qualitative analysis which, carried out in a solely chemical manner, has become less important.[2]

In the general chemistry laboratory at the University of Marburg the beginners study the reactions of the metals in test tubes in the usual way. However, the chemicals used are not necessarily the most selective and sensitive reagents for the detection of the particular metal ion. Instead, the tests reveal the reactions of the element with simple inorganic precipitating agents, the chemistry of the complex compounds it forms, and the inorganic reactions in which the element differs from those of the same group of the periodic system or from others similar in chemical behavior.

It is an old experience of the chemistry teacher that the important reactions of the elements impress the student only if they are performed repeatedly. Also, skill in the most important practical chemical methods like precipitation, decantation, washing, dissolving, etc., is obtained only by repetition. What was formerly the pedagogic task of qualitative analysis is now taken over by the preparative separations mentioned here. These preparative separations are essentially a reinstatement of the classical qualitative analytical course of separation. The usual classification of analytical groups is therefore retained.

The Separations

The student obtains 150–200 mg of a soluble mixture of metal salts or 5 ml of a solution containing 15–20 mg each of different metal ions. He is supposed to separate the metals in form of precipitates that are sparingly soluble. First the HCl group is precipitated with dilute hydrochloric acid and separated by centrifugation. The precipitate is washed and the lead chloride is extracted with hot dilute hydrochloric acid in the usual way and then treated with chlorine or bromine water. Mercury present as Hg_2Cl_2 dissolves as $HgCl_2$ while silver chloride remains undissolved and is separated by centrifugation and washed. The centrifugate is boiled briefly to remove the halogen, and sodium hydroxide is added. HgO precipitates which is separated and washed.

The H_2S group is precipitated from the centrifugate of the HCl group and separated into the arsenic and copper groups in the usual manner. The next steps also follow closely the classical analytical procedures. However, it is not sufficient to recognize copper on the basis of the blue color of the ammonia complex. It has to be precipitated as CuSCN and separated by centrifugation. Tin, after reduction by iron or nickel, is not simply identified by its reducing properties but is precipitated as SnS and separated.

In the $(NH_4)_2S$ group more pronounced deviations of the separations suggested here from the classical procedures are necessary. After precipitation as hydroxides, iron and titanium are separated by extracting the HCl solution with methyl isobutyl ketone. Nickel is precipitated as $[Ni(NH_3)_6](ClO_4)_2$ and the cobalt, dissolved in the filtrate as pentammine aquo complex, is precipitated as the sulfide. The separation in the

[1] Mahr, C., *Angew. Chem.*, **67**, 712 (1955).
[2] Strong, Frederic E., J. Chem. Educ., **34**, 400 (1957).

ammonium carbonate group finally is based on the usual precipitations as barium chromate, strontium sulfate, and calcium oxalate. In general, a separation of the alkali metals is not required. It can be carried out relatively easily by extracting lithium as chloride with ethanol and by precipitating potassium as perchlorate.

After performing the separations the student hands in the centrifuge tubes which contain each of the precipitates of the metal ions present in the original mixture. The instructor judges the student's performance on the basis of purity, color, form, and quantity of the precipitates. This is done more easily and correctly than is possible by reading the usual written lab reports.

Other Advantages

In these separations, the student is asked to carry out only inorganic reactions. He cannot apply in advance color tests with organic reagents, the nature of which he cannot yet understand. The separation problem has the same wide scope in varying the components as the former qualitative analysis. The student has much choice in selecting his own methods for separation, as has been recommended recently for qualitative analysis.[3] For several semesters, this method has proved to be quite successful in the department of chemistry at the University of Marburg. The separations are carried out according to the textbook of the author[4] and, with the usual amount of training, they yield good results in independent work and critical thinking. Also they effectively prepare the students for qualitative analysis which can now be taught in a completely modern form. In the book mentioned above[4] the description of the separations is therefore followed by a discussion of the new techniques of qualitative analysis, the semimicro method with special reagents and the analysis by paper chromatography.

Acknowledgment

The author wishes to thank Professor K. W. Starke of the University of Marburg for help received during the translation of this article.

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Detection of Phosphate in the Presence of Arsenate

There are two procedures[1,2] for the detection of phosphate in the presence of arsenate. In the first,[1] both anions are precipitated with magnesia mixture, arsenate reduced to arsenite by SO_2 in acidic medium, and the arsenite eliminated by precipitation as As_2S_3. Phosphate is then detected in the centrifugate with ammonium molybdate. The procedure is laborious. In the second,[2] ammonium molybdate-tartaric acid reagent is employed, as tartaric acid prevents the interference due to arsenate. This procedure is rapid.

The procedure here described is based on the observation that magnesium ammonium arsenate dissolved in dilute HCl gives a brown precipitate in the cold on the addition of sodium dithionite, and the centrifugate does not give a positive test for arsenate with ammonium molybdate and nitric acid, even on boiling for minutes. The brown precipitate is tritarsenic sulfide, As_3S.[3,4] Since phosphate remains unaffected, it can be detected with ammonium molybdate after the elimination of arsenate.

Procedure

Precipitate the two anions with magnesia mixture, following the directions given.[1] Detect arsenate by the usual tests, and phosphate by the procedure described.

Suspend 20 mg of the ppt in 0.5 ml of water and add one or two drops of 5 *N* HCl to dissolve the ppt. Add just one drop more, followed by 25–50 mg of sodium dithionite. Wait one minute, and centrifuge. Reject the residue and transfer the centrifugate into a semimicro boiling tube. Add 1 ml of concentrate HNO_3. Sulfur gets precipitated. Boil till sulfur is oxidized and the liquid becomes clear. Finally add 1 ml of ammonium molybdate and heat to boiling. Yellow ppt or color shows the presence of *phosphate*.

[1] VOGEL, A. I., "A Text-Book of Macro and Semimicro Qualitative Inorganic Analysis," 4th ed., Longmans, Green and Co. Ltd., London, 1954, p. 479.
[2] *Ibid.*, pp. 388, 389.
[3] MELLOR, J. W., "A Comprehensive Treatise on Inorganic and Theoretical Chemistry," Longmans, Green and Co. Ltd., London, 1946, Vol. 10, p. 173.
[4] *Ibid.*, pp. 183, 184.

What Is It? Sure It Is!

A student placed a test tube of concentrated hydrochloric acid and another of concentrated ammonia side by side in a block and placed the block in his locker drawer. The next week he took them out and found the result pictured on page 399.

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Non-Cyanide Detection of Cadmium in the Presence of Copper

In the classical procedure (*1*), both copper and cadmium are bound into their complexes with KCN. The copper complex has a lower instability constant than the cadmium complex has, so that only the sulfide of the latter ion is precipitated by H_2S. The highly poisonous nature of KCN has stimulated the search for non-cyanide procedures. A list of non-cyanide methods is given in "Literature Cited" (*1–11*).

This communication describes six of nine new non-cyanide procedures worked out by the author. The other three will be published in *School Science Review*.

The semimicro procedure is suggested here; it is presumed that the directions given in reference (*1*) are followed. Also it is taken for granted that in every case, copper is to be detected as the red-brown ferrocyanide, for which purpose a part of the solution of ammines of copper and cadmium is acidified before the addition of potassium ferrocyanide.

(I)

It is the author's observation that ammonium oxalate, in neutral medium, precipitates cadmium and complexes copper preferentially into a soluble light blue complex. The nature of the complex could not be ascertained from the literature accessible to the author. Perhaps a stable anion, similar to the one formed by aluminum and oxalate ions (*12*), is formed. The author invites readers' comments.

Reagent: 0.5 *N* ammonium oxalate.

Acidify six drops of the blue solution with 5 *N* H_2SO_4 (color changes to pale blue). Add two drops more, followed by 0.2–0.3 g $BaCO_3$ (*13*). Heat well to expell all CO_2 and centrifuge. Reject the residue and make sure that the centrifugate is not acidic toward litmus. Now add 8–10 drops of the reagent to 6 drops of the neutral solution, shake well, and centrifuge again. Any ppt shows *Cd* is present. Reject the centrifugate and wash the residue with 1 ml of cold water. Transfer one or two drops of 10% solution of Na_2S. Yellow ppt confirms the presence of *Cd*.

(II)

This procedure is based on the observation that in neutral medium, copper gets preferentially complexed by ammonium carbonate as well as sodium bicarbonate. Again the author could not ascertain the composition of the complexes. In practice neutralization of the acid is unnecessary. The reagent does the neutralization as well.

Reagent: 4 *N* ammonium carbonate *or*, 8% solution of sodium bicarbonate.

Acidify six drops of the blue solution with 5 *N* HCl, and add 6–8 drops of either of the two reagents. Shake well and let stand 2–3 minutes. Follow the directions given under procedure (I). Once again, yellow ppt confirms the presence of *Cd*.

(III)

In the presence of sulfurous acid, potassium iodide reacts with copper(II) salt according to the equation (*14*),

$$2\ CuSO_4 + 2\ KI + SO_2 + 2\ H_2O \rightarrow 2\ CuI + K_2SO_4 + 2H_2SO_4$$

0.2 *N* KI dissolves 0.000157 mole of copper(I) iodide per liter (*15*). Thus excess of potassium iodide should be avoided. The acid is neutralized by barium carbonate.

Reagent: 10% solution of potassium iodide.

Acidify six drops of the blue solution just to neutrality. Add three drops each of saturated H_2SO_3 and the reagent. Add 25 mg of $BaCO_3$, shake, boil, and centrifuge. Reject the residue and expel SO_2 from the centrifugate by heating in a boiling water bath for 2–3 minutes. Finally add one or two drops of 10% Na_2S solution to the SO_2-free centrifugate. Yellow ppt shows the presence of *Cd*.

(IV)

If sodium thiosulfate solution is added to the blue solution of copper and cadmium ammines, the blue color is discharged on heating. On heating to dryness, a black residue, presumably due to the formation of copper(I) sulfide, is obtained. Under the circumstance cadmium ion remains unaffected and gets extracted with water readily.

Reagent: 10% solution of sodium thiosulfate.

Transfer six drops of the blue solution into a semimicro boiling tube (or a small casserole), followed by three or four drops of the reagent. Heat to dryness. Heat for several seconds more. Let cool and extract the residue with 0.5 ml hot water. Reject the black Cu_2S; add one or two drops of 10% solution of Na_2S to the water extract. Yellow ppt is due to *Cd*.

(V)

In mildly acidic medium, ethanolic "oxine" (8-hydroxy quinoline) precipitates only copper(II) "oxinate" (*16*), so that cadmium can be detected in the centrifugate. The procedure described is superior to the Boyd and Easley (*8*) procedure, which involves extraction of copper(II) ammine with chloroformic "oxine" after *p*H adjustment.

Reagent: 2% ethanolic solution of "oxine."

Acidify four drops of the blue solution of ammines with 10% acetic acid, and add six drops more of the acid. Now add 4–6 drops of the reagent, heat and centrifuge. Reject the residue and add one or two drops of 10% solution of Na_2S to the centrifugate. Yellow ppt reveals the presence of *Cd*.

(VI)

Zinc mercaptide of toluene-3,4-dithiol (ZDT) precipitates copper(II) in solutions as highly acid as 6*N* HCl. Cadmium, in contrast, is not precipitated if the acidity exceeds that of 1*N* HCl (*17*). This affords another method for the detection of cadmium in the presence of copper.

Reagent: ZDT powder.

Add 6–8 drops of 5 *N* HCl to six drops of the blue solution, followed by 25 mg of the reagent. Heat in a boiling water bath for about two minutes and centrifuge. Reject the black residue and dilute the centrifugate to 1 ml. Add one or two drops of 10% solution of Na_2S. Yellow ppt confirms *Cd*.

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(14) *Ibid.*, p. 202.

(15) *Ibid.*, p. 204.

(16) Vogel, A. I., "A Text-Book of Quantitative Inorganic Analysis," 2nd ed., Longmans, Green & Co. Ltd., London, **1951**, p. 373.

(17) Clark, R. E. D., and Neville, R. G., J. Chem. Educ., **36**, 390 (1959).

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Benzenoid Nitro-Compounds as Organic Reagents for Tin(II)

Tin is an element easily missed by qualitative analysis students using conventional procedures. The main difficulty is that the reaction with mercury (II) chloride (*1*) is a general test for reducing agents and not specifically for tin(II) ion. Furthermore the test is inconclusive for small concentrations of tin.

Welcher and others (*2*, *3*, *4*) used naphthol yellow-S as a reagent for tin. Naphthol yellow-S in the presence of potassium hydroxide gives a blood-red solution for a wide range of concentrations. The summary of results with possible interfering cations is given in Table 1.

The reaction with naphthol yellow-S as given by Welcher appears to be an oxidation-reduction reaction involving the strong reducing power of the tin(II) ion and probably a chelation of the reduced nitro compound with the tin(IV) ion. A blank solution of naphthol yellow-S and potassium hydroxide gives a whitish-yellow precipitate and a yellow solution. This precipitate appears to be analogous to the one which accompanies the characteristic red color with the tin(II) ion.

To determine whether the sulfonic group and the naphthalene ring were essential for the positive reaction of naphthol yellow-S, 2,4-dinitrophenol was tested. It was found that 2% solutions of naphthol yellow-S and 2,4-dinitrophenol reacted in exactly analogous fashion; a blood-red solution was obtained with only tin(II) ion; none of the other group II elements reacted; iron(III) ion produced a masking orange-brown precipitate. A summary of results using 2,4-dinitrophenol as an indicator are given in Table 2.

2,4-dinitrophenol differs from naphthol yellow-S in that it does not form a precipitate with potassium hydroxide in a blank solution. Hydrochloric acid removed the red color from the solution.

When equal portions of 0.5 *M* tin(II) chloride and 2,4-dinitrophenol were allowed to stand at room temperature for three to five minutes, a colorless crystalline material precipitated. The filtrate of this mixture gave the characteristic cherry-red color reaction upon addition of base. The precipitate, however, produced only a yellow solution with potassium hydroxide. The precipitate was found to be entirely organic, containing no metallic ions.

Table 1. Reaction of 2% Naphthol Yellow-S with Cations (0.5 M)

Sn^{+2}	Blood red
Sb^{+3}, As^{+5}, As^{+3}, Sn^{+4}, Cd^{+2}	White ppt
Hg^{+2}	Bright yellow ppt
Hg_2^{+2}	Yellow-orange ppt
Pb^{+2}	Orange ppt
Bi^{+3}	White-yellow ppt
Cu^{+1}, Cu^{+2}	Grass-green ppt
Fe^{+3}	Brown ppt
Fe^{+2}	Orange-red ppt

The experimental work described in this paper was supported by a research grant from the Research Corporation of New York.

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Table 2. Reaction of 2% 2,4-Dinitrophenol with Cations (0.5 M)

Sn^{+2}	Blood red
Sn^{+4}, Sb^{+3}, As^{+5}, As^{+3}	White ppt
Hg^{+2}, Hg_2^{+2}	Yellow ppt
Cu^{+1}, Cu^{+2}	Green ppt and solution
Pb^{+2}	Yellow solution
Bi^{+3}, Cd^{+2}	White ppt, yellow solution
Fe^{+3}, Fe^{+2}	Orange-brown ppt
Co^{+2}, Cr^{+3}	Green solution
Ni^{+2}	No reaction

None of the other ions in the tin-arsenic group gave interfering reactions. The use of potassium hydroxide rather than ammonia as a base overcame the difficulty of the blue ammonia complex of copper if copper were accidently drawn into the arsenic subgroup solution. Most colored ions interfere to some degree, although in most cases to a very small extent.

The following simple procedure is suggested as a definite test for the presence of tin(II) ions in liquid unknown samples such as used in the conventional qualitative analysis course:

One ml of unknown is added to 2 ml of a 2% aqueous solution of 2,4-dinitrophenol; 5 ml of 0.1 *M* solution of potassium hydroxide is then added. The formation of a blood-red solution indicates the presence of tin(II) ions.

A series of experiments was performed with meta-dinitrobenzene using the above procedure. A 2% solution in isopropyl alcohol gave a royal blue solution, which is specific for tin(II) ion.

When 2 ml of a 2% (saturated) solution of meta-dinitrobenzene was added to 0.5 *M* tin(II) chloride, a yellow solution formed which darkened in a matter of seconds. Upon addition of the base, a royal blue solution was produced in addition to a dark precipitate. The color of the precipitate varied from a light gray to dark brown, although the blue solution always formed. The blue color changed in a matter of minutes to a mud brown. The colored material was insoluble in benzene but dissolved in *n*-butyl alcohol and appeared to be easily oxidized in air.

The other ions of the group show no interfering color reactions. Most colored ions interfere as was the case with 2,4-dinitrophenol. A summary of results with the compound is given in Table 3.

Both 2,4-dinitrophenol and meta-dinitrobenzene are more easily available and cheaper than naphthol yellow-S. Both reagents can be easily prepared in the laboratory by a student knowing the rudiments of organic preparatory chemistry.

Table 3. Reaction of 2% Meta-Dinitrobenzene with Cations (0.5 M)

Sn^{+2}	Royal blue
Sn^{+4}	No reaction
Sb^{+3}, As^{+5}, As^{+3}, Pb^{+2}, Bi^{+3}, Cd^{+2}	White ppt
Hg^{+2}	Yellow ppt
Hg_2^{+2}	Orange ppt
Cu^{+2}	Sky-blue ppt
Cu^{+1}	Green ppt
Fe^{+3}	Orange-to-brown ppt
Fe^{+2}	Mud-brown ppt
Ni^{+2}	Pink solution

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(4) Tenney, H. M., and Long, H. J., J. Chem. Ed., **13**, 82 (1936).

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A New Qualitative Reagent for Aluminum

The laboratories of Walla Walla College and Oregon State College have used arsenazo, 3-(2-Arsonophenylazo)-4,5-dihydroxy-2,7-naphthalenedisulfonic acid (available from Eastman Organic Chemicals, Rochester 3, New York), in the elementary qualitative analysis classes as a confirmatory test for aluminum. This reagent seems to be quite superior to Aluminon. In one course where 19 unknowns contained aluminum only one student missed it. Another class of 80 students reported that no students missed aluminum in the general unknowns.

No change in procedure is required for the use of this reagent. Follow the regular procedure for Group III down to precipitation of $Al(OH)_3$ and centrifuge off. Dissolve the precipitate in dilute acetic acid (*p*H should be 3–7). Add 2 drops of arsenazo reagent (0.001 *M* solution). If aluminum is present a reddish-purple color results. If no aluminum is present the solution becomes orange-colored.

The arsenazo reagent is water soluble, consequently the reagent solutions are easily prepared. The solutions are stable indefinitely and the purple aluminum complex is also stable.

There are two possible sources of error:

(1) If copper slips through and appears in Group III it will give a purple color with arsenazo. This is not very likely to happen, however.

(2) If the solution at the time of the confirmatory test for aluminum is very dilute, the faint purple color might be missed.

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Chloride, Bromide, and Iodide Determination

There is a need for a relatively simple qualitative scheme of separation and identification for chloride, bromide, and iodide, particularly when all three ions are present or when halide, sulfide, cyanide, and thiocyanate ions may interfere. Most analysis schemes now in use involve the precipitation of the silver halides along with any silver cyanide, silver sulfide, and silver thiocyanate. The precipitate is treated in various ways to put the ions back into solution and then tests are made for the anions.

The simple test described here is relatively free from interfering ions and may be used to identify halides in both inorganic and organic qualitative analyses. The test may be made, without interference, on a solution made up from 1 ml each of 0.1 *M* solutions of the following anions: $(CO_3)^{-2}$, $(SO_4)^{-2}$, $(SO_3)^{-2}$, $(PO_4)^{-3}$, $(F)^{-1}$, $(C_2C_4)^{-2}$, $(Cr_4)^{-2}$, $(AsO_4)^{-3}$, $(AsO_2)^{-1}$, $(BO_2)^{-1}$, $(CN)^{-1}$, $(CNS)^{-1}$, $(S)^{-2}$, $(C_2H_3C_2)^{-1}$, $(NO_3)^{-1}$, $(SH)^{-1}$, and $(HCO_3)^{-1}$.

Ferricyanide and ferrocyanide ions cause interference and must be removed by precipitating them with cadmium acetate. The centrifugate is used for testing.

Procedure

Put four drops of the unknown solution (free of ferricyanide and ferrocyanide) in a 4-in. test tube. If the unknown is basic, add concentrated HNO_3 by drops with stirring until the unknown is just acid. Then add two drops of concentrated HNO_3, 0.5 ml CCl_4, and shake the mixture vigorously. A pink-to-violet coloration of the CCl_4 layer indicates the presence of iodine. No coloration of the CCl_4 layer indicates the absence of iodine. If iodine is present, remove the CCl_4 layer by means of a pipet, add one additional drop of concentrated HNO_3 and again extract with 0.5 ml CCl_4. Continue the extractions until the CCl_4 layer is colorless or a very light pink.

To the aqueous layer add 2 ml concentrated HNO_3. Add 0.5 ml CCl_4 and shake vigorously. A reddish-to-straw-yellow coloration of the CCl_4 layer indicates bromine. No coloration of the CCl_4 layer indicates the absence of bromine. Remove the CCl_4 layer by means of a pipet and extract the aqueous layer again with 0.5 ml CCl_4. Continue the extractions until the CCl_4 layer is colorless.

If there is some coloration of the aqueous layer after the bromine extraction, boil the solution until it becomes colorless. Otherwise, boil the solution for approximately 30 seconds. If thiocyanate ion is absent, the boiling is unnecessary.

To the colorless aqueous solution, add 2–3 drops of a 5% aqueous silver nitrate solution. An immediate formation of a white precipitate indicates chloride. No cloudiness or precipitate indicates the absence of chloride.

The same procedure may be applied to a sodium fusion filtrate obtained from the fusion of metallic sodium and organic compounds. Cyanide, sulfide, sulfate, fluoride, and phosphate did not interfere with the analysis of a sodium fusion filtrate containing chloride, bromide, and iodide ions.

If thiocyanate is absent and the iodide concentration is low, as indicated by a light pink coloration of the CCl_4 layer, it is not necessary to remove the CCl_4 layer. The following procedure may be used:

To four drops of the unknown solution in a 4-in. test tube add 2 drops of concentrated HNO_3 and 0.5 ml CCl_4. Shake well. A light pink CCl_4 layer indicates iodide. Add 2 ml of concentrated HNO_3 to the mixture and shake well. A change in color from a light pink to a reddish-yellow or a straw yellow indicates the presence of bromine. No change in color indicates the absence of bromide. Remove the CCl_4 layer and extract again with 0.5 ml CCl_4. If the CCl_4 layer is practically colorless, add 2–3 drops of a 5% aqueous silver nitrate solution to the aqueous layer. An immediate formation of a white precipitate indicates the presence of chloride.

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A Simple Test for Nitric Acid Fumes

For the evaporation of a solution of mixed nitric and sulfuric acids to the complete decomposition of the nitric acid, procedures usually instruct the student to evaporate "until dense, white, choking fumes of sulfur trioxide can be seen." Recognizing sulfur trioxide fumes is often a problem for students; frequently evaporations are stopped too soon.

An effective test for detecting the point at which all HNO_3 is gone is as follows: The beakers containing the evaporating solutions are loosely covered by watch glasses supported on glass "elbows." A drop of the condensate on the bottom of the watch glass is touched to an ordinary kraft paper towel. The spot turns brown if any nitrogen oxides or nitric acid are present in the drop. After all the nitric acid is gone, the spot on the towel will remain colorless.

The lower limit of nitric acid concentration detectable has not been determined. Where mere removal of nitric acid is appropriate, there is no need to continue the heating until sulfur trioxide fumes appear; rather, the heating can be discontinued when a negative "towel" test is obtained.

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Rapid Detection of Cations of Copper Group

As an alternative to the conventional cation Group II separation and subsequent schematic analysis, the following proposal permits rapid detection of the ions normally present in the copper subgroup. The procedure is based on the following evidence:

Mercury, from an acid solution, is deposited on a strip of copper foil after brief contact;[1] whereas arsenic, antimony, and bismuth deposit very slowly. Precious metals interfere.

Of the phosphates of mercury, lead, bismuth, copper, and cadmium, only that of bismuth is sparingly soluble in dilute mineral acids.[2] As little as 20 micrograms of bismuth is detectable in 5 *N* nitric acid.

An excess of concentrated ammonium hydroxide precipitates lead and bismuth and complexes copper and cadmium as tetrammines.

Sodium dithionite ($Na_2S_2O_4$) reduces copper salts to metallic copper in ammoniacal solution.[3,4]

[1] Gaston, Charlot, et al., "Rapid Detection of Cations," 2nd ed., Chemical Publishing Co., Inc., New York, **1954**, p. 30.
[2] Vogel, A. I., "Macro and Semimicro Qualitative Inorganic Analysis," 4th. ed., Longmans, Green & Co., Inc., London, **1954**, p. 233.
[3] Vogel, A. I., *op. cit.*, p. 415.
[4] Clark, R. E. D., and Neville, R. G., J. Chem. Educ., **36**, 393 (1959).

Procedure

The sample should be homogeneous and contain approximately 10 mg each of Hg^{+2}, Pb^{+2}, Bi^{+3}, Cu^{+2}, and Cd^{+2} ions.

Mercury: After the removal of the Group I cations, place one drop of the slightly acidified sample solution on a strip of polished copper foil. After several seconds, rub the spot under water. A white amalgam indicates *Hg*.

Lead, Bismuth, Copper, and Cadmium: Precipitate the ions of Group II with H_2S or thioacetamide. Separate, by conventional means, the Group II precipitate into the copper and arsenic subgroups. Treat the copper subgroup (as sulfides) with 20–25 drops of 5 *N* HNO_3, warm, and centrifuge. Discard any residue. To 6 drops of the centrifugate add 2–3 drops of 2 *N* $(NH_4)_2SO_4$. A white precipitate shows the presence of *Pb*. To 6 drops of the centrifugate add 2 drops of 5 *N* HNO_3 and 2–3 drops of 1 *N* $(NH_4)_2HPO_4$. A white precipitate shows the presence of *Bi*. Add concentrated ammonium hydroxide in excess to the remainder of the centrifugate. Centrifuge and discard the residue. If the ammoniacal centrifugate is blue, copper is present. The presence of copper is confirmed by adding 5 *N* acetic acid to 6 drops of deep blue centrifugate until the color changes to a light blue; followed by 2 drops of 0.5 *N* potassium ferrocyanide. A red-brown precipitate shows the presence of *Cu*. To 6 drops of the ammoniacal centrifugate add about 25 mg of $Na_2S_2O_4$ if copper is present. Boil, centrifuge, and discard the residue of metallic copper. Repeat the addition of $Na_2S_2O_4$ if blue color remains. To the clear or yellow centrifugate add H_2S, or thioacetamide (followed by warming). A yellow precipitate shows the presence of *Cd*. An excess of $Na_2S_2O_4$ and a source of sulfide ions is to be avoided as the former decomposes the latter into colloidal sulfur and water.

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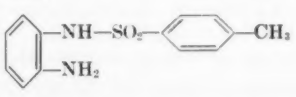
A Specific Qualitative Reagent for Copper

The separation and confirmation of copper, particularly in samples containing cadmium, often cause trouble in the conventional qualitative analysis scheme. We are here suggesting the use of a specific precipitant for separation and confirmation of copper-(II) ions.

Billman[2] has used *o*-(*p*-toluylsulfonamide) aniline, (T-sulfonamidine)



for the quantitative determination of copper in brass.

[1] This work is taken in part from the senior independent study of Frederick Burton.
[2] Billman, J. H., Janetos, N. S., and Chernin, R., *Anal. Chem.*, **32**, 1942 (1960).

The precipitation of copper with T-sulfonamidine is quantitative over the *p*H range 6.2 to 8.5.

A solution suspected of containing copper is adjusted to a *p*H of about 7 (Hydrion paper) by the addition of HCl or NH_3. The hydrolysis products of cations such as bismuth, mercury, antimony or silver are removed by centrifuging. The resulting solution is treated with a few drops of 1% T-sulfonamidine; the appearance of a green precipitate confirms the presence of copper. Solutions as dilute as 0.001 *M* in Cu^{++} may be detected using this reagent but those less than 0.01 *M* are slow to precipitate and thus require heating. The separation of mixtures of copper and cadmium may be easily performed in this way. If cadmium is present in the resulting solution, yellow CdS may be precipitated by thioacetamide. None of the cations most commonly present in a qualitative analysis scheme interfere with this test.

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Ion Exchange Separation of Cadmium

The procedure that is generally employed by the various schemes of qualitative analysis for the identification of cadmium ion consists of the precipitation of yellow cadmium sulfide at the end of the analysis for the particular group or sub-group containing the cadmium ion. Successful performance of the test thus depends upon the quantitative removal of all other ions prior to the test, and it is not uncommon for students to obtain a black precipitate as a result. The following ion exchange separation is an alternative to the usual advice of "go back and repeat the analysis." It has been used successfully with both a hydrogen sulfide scheme[1] and a non-sulfide scheme[2] of analysis.

The ion exchange resin to be used is a strong-base type anion exchanger (e.g., Dowex-1) which has been conditioned to the bromide ion salt form. With the hydrogen sulfide scheme, the solution which is used for the test is the solution obtained from treating the copper sub-group precipitate with nitric acid. With the non-sulfide scheme, it is convenient to use a solution containing the entire group.

Ten drops of the appropriate unknown solution are added to about one gram of the air-dried resin contained in a test tube, then twenty drops of 0.1 *M* potassium bromide solution are added. The mixture is agitated vigorously in the test tube with a stirring rod for thirty seconds, after which the resin particles are allowed to settle. The supernatant liquid is drawn off with a small pipet or medicine dropper and discarded. Another twenty drops of the potassium bromide solution are added and the procedure is repeated. The resin should be washed in this manner with 0.1 *M* potassium bromide for a total of five or six times. After the last wash portion has been withdrawn and discarded, the resin is washed thoroughly but only once with concentrated ammonium hydroxide. A few drops of the ammoniacal supernatant liquid may be withdrawn and tested for the presence of cadmium ion by the addition of a drop or two of sodium sulfide solution.

The separation is simple and requires only a few minutes to perform. Furthermore, it serves as an introduction to a modern technique at a time when it will be most appreciated. After a brief explanation of the fundamental principles of anion exchangers, the elucidation of the chemistry involved in the above separation makes an excellent exercise for the student.

[1] Stubbs, Morris F., and Jones, W. Norton, Jr., "Laboratory Exercises in General Chemistry," Wm. C. Brown Co., Dubuque, Iowa, 1954, p. 235.

[2] West, Philip W., and Vick, Maurice M., "Qualitative Analysis and Analytical Chemical Separations," 2nd ed., The Macmillan Co., New York, 1959, p. 52.

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A Simple Test for the Detection of Phosphorus in Organic Compounds

We have been experimenting with the introduction of phosphorus-containing compounds in our course in the identification of organic compounds. The following qualitative test for phosphorus has been developed.

During the usual sodium fusion procedure,[1] a piece of filter paper, previously moistened with silver nitrate solution, is quickly placed over the mouth of the test tube after the sample has been dropped into the sodium vapor. The immediate production of a jet-black color on the filter paper constitutes a positive test for phosphorus.

Phosphorus-free compounds which contain nitrogen, iodine, or sulfur cause darkening to gray or brown, but these colors should not be interpreted as positive tests. The test papers may become black when allowed to stand, but only an *immediate* change to black indicates the presence of phosphorus. In the sodium fusion of more highly oxidized compounds such as phosphate esters, it is important that sucrose be present; otherwise the test may be inconclusive.

The color production is assumed to depend upon the evolution of phosphine or phosphorus which in turn reduces the silver ion to the free metal.[2]

This test has the advantage that no additional oxidative step is necessary either on the unknown or on the sodium fusion test solution in order to detect phosphorus as phosphate. Students have used the test successfully on a wide variety of compounds ranging from phosphines to phosphate esters.

[1] Shriner, Ralph L., Fuson, Reynold C., and Curtin, David Y., "The Systematic Identification of Organic Compounds," 4th ed., John Wiley and Sons, Inc., New York, 1956, p. 57.

[2] Van Wazer, John R., "Phosphorus and Its Compounds," Interscience Publishers, Inc., New York, 1958, pp. 132, 133.

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Translating and Re-Jargonizing the Little-Languages of Science

It is trite to say that as science blossoms in splendor the likes of which we have never known, a hungry non-science public waits for each and every new development. Finding a way to satisfy the public appetite for science is, however, a very real problem. Bridging science and non-science is becoming more and more difficult, for as the language of science moves in one direction, the language of non-science moves in the other.

On the one side are the many jargons and little-languages of science; on the other side are the many slangs and little-languages of non-science. Somewhere in between is what most of us would prefer—good, simple English.[1]

The professional scientist, wittingly or not, has set an example or at least has followed a trend (one might even call it a recurring fad). He speaks and writes in one or more of the different varieties and species of the often-remote little-language of *sciencese*.

The problem, therefore, is not only communication of science with non-science, but also intercommunication among the various specialties within science. The latter is essentially no different from the former, for when one is illiterate in a given science argot it matters little whether he is a scientist.

The problems in translating and re-jargonizing can be said to derive from two maxims:

(*A*) *Non-communicated science is non-existent science.*

(*B*) *Non-communicable science is non-existent science.*

A recent editorial in THIS JOURNAL[2]—discussing C. P. Snow's "The Two Cultures and the Scientific Revolution"[3]—handily set out the gist of things:

> It is easy to talk in generalities about society and forget that we are individuals who can do something about it. It is not enough to smugly point out that there are more scientists who know who Job or Hamlet was than there are literary men who know what the second law of thermodynamics is or why Libby won the Nobel prize. *Rather there is an increasingly pressing personal obligation to tell the non-scientist what science is doing. Each must do this in his own way; there is no formula to follow. The obvious ingredients are a thorough understanding of what the essential concepts are and a willingness to abandon the jargon of the professional scientist when doing it.* [italics added]

Examples of science lingo—sciencese—are familiar to all of us. In the semantic hierarchy we may take the following for examination:

> . . . existentialism-oriented within the contemporary American socio-political macro-module. . .

Translated into simple English this may become:

> . . . hedonistic (or materialistic) trend in present American life. . .

Fully re-jargonized for the man-in-the-street this becomes:

> . . . gut-and-buck jag . . .

Crude, coarse, and undignified? Perhaps. But the last gets the point over and carries a very plain message which is brief, to the point, and easily remembered.

From Jargon to Jargon

The kinds of sciencese—their number has never been estimated—must be truly enormous, involving dead and dying jargons as well as the firmly established and the recently born. Borrowings and re-definitions are characteristic of sciencese. *Extra-mural borrowings:* sunshine unit, aeon, barn, bit, pot, dope, jerk, and the names of things, places, persons, and events. *Intra-mural borrowings:* symmetry, countdown, fission, fusion, field, energy, space, radio, ray, and wave (used jargon-wise at least several hundred different ways). When science borrows from itself—and makes great leaps in re-definition—it engenders difficulties for both science and non-science.

Certain science jargons amount to cryptic slang, e.g., the little-languages of cybernetics (we have psycho-cybernetics), the variegated schools of psychologic and psychiatric thought and, especially, the argotese arising from the hybridization of several little-languages, e.g., magnetohydrodynamics, biomedical electronics, control engineering, computers, avionics, bionics, psychopharmacology, and paraweaponry.[4]

In some cases, often unfortunate ones, a true science code is approached or actually attained, as in advanced military and space science and technology (readers acquainted with classified literature often encounter "projected thinking" reports where a peculiar code-like lingo completely defeats the purpose and makes the document unintelligible).

The titles of scientific and technical papers show the ever-increasing trend to science code and cryptic slang based upon a huge preponderance of clippings, acronyms, tip-terms, and the like. Onomatopoetic or echoic synthetics and coinages are frequent. The reader may find himself engaged in mental gymnastics trying to guess the specific meaning or particular discipline of the following:

E-Type X Circulator
The S Plant
The N Process

[1] This paper contains built-in examples of the subject at hand. The term "little-language" is used more loosely here than that defined by WENTWORTH, H., AND FLEXNER, STUART B., "Dictionary of American Slang," Th. Y. Crowell, New York, 1960.

[2] J. CHEM. EDUC., **37**, 609 (1960).

[3] SNOW, C. P., "The Two Cultures and the Scientific Revolution," Cambridge Univ. Press, New York, 1959.

[4] Any day now I expect to see in print the terms geogeriatrics, astrohormology, and psychocosmochemistry.

Effects of Mountains with Smooth Crests on Wave Propagation
Some Remarks on the Spreading Reaction
Constant-S Equalizers
Are Statistical Life-Testing Procedures Robust?
Is Horizontal Education Lying Down?
Experimental Immersion Leg
Role of Cleft Palate Team
L. E. Prep Technique
/CTS/Unrestive Feedback of Psychobionic Scission in Boolianized Agate State Barrier/CTS/[5]

The examples just given were picked at random. One only has to glance through a current issue of *Chemical Abstracts, Biological Abstracts, Nuclear Science Abstracts,* or any other similar periodical—especially bibliographies of government research reports—for verbal do-it-yourself kits.

When bred from science and law the offspring can become a true semantic mutant: a case in point is forensic sciencese and its numerous species. For example, the little-languages set out in the chemical patent are very apt to be completely alien to chemists not directly engaged in industrial or applied research. Those in strictly theoretical or "pure" chemical research often find chemical patent lingo as baffling as *pentagonese.*

Scientific pentagonese has dramatically changed since Sputnik-I. It is probably the fastest moving and "farthest out" of all lingos, beatnik and jive talk notwithstanding. This is sad commentary on a kind of jabberwocky which, because of its enormous significance, should be among the clearest of little-languages.

Those slangs which are the fastest moving—and get farther and farther out—resemble fads in that they are most likely to die quickly, be replaced, and be forgotten sooner (however, depending upon their coverage and impact, they often contribute to the body of the more static little-languages). Scientific pentagonese exemplifies how, within a period of a few years, a succession of dead and dying lingos is built.

What is said above applies also to a vigorous and growing family of slangs as yet unnamed: those having to do with politics and science [and these would seem to have little to do with the more orthodox little-language(s) of political science (a misnomer)]. These working slangs are not now widely known or understood because of their newness, but they will be of extraordinary importance during the coming years. When they become so firmly embedded as to work their way into the language of the law they will really have come into their own.

The mixed-meaning terms of science itself would fill volumes and, indeed, have done so (e.g., specialized dictionaries, glossaries, and encyclopedias). An interesting example of what goes into science's little-languages comes from my own field, that of luminescence. We have, for example:

Fluorometry	Phosphorometry
Fluorimetry	Phosphorimetry
Photofluorometry	Phosphorophotometry
Photofluorimetry	Photophosphorimetry
Spectrophotofluorometry	Spectrophotophosphorometry
Photospectrofluorimetry	Photospectrophosphorimetry
Spectrofluorophotometry	Spectrophosphorophotometry
Fluorispectrophotometry	Spectrophotophosphorimetry
Photofluorospectrometry	Phosphorospectrophotometry

[and some few more]

[5] Hypothetical classified document: /CTS/ = Cosmic Top Secret (NATO). The other titles are real.

Now, which of these has to do with fluorescence? Which with the measurement of fluorescent substances? Which with fluorine chemistry? Which with the analysis of fluorine? Which with phosphors and the measurement of their spectral emission? Which with the solid-state physics of phosphors? Which with phosphorus chemistry? Which with phosphorus determinations generally? Which with phosphorus determinations spectrophotometrically? And so on.[6]

Semantic Equations

Actually, each of the previous terms is a *semantic equation* with different solutions, depending upon the discipline. The puzzle—and sometimes the danger—for the unwary science translator is to find out as best he can which term means what. As the same or similar terms become more firmly embedded in the matrices of different little-languages, the problem of correct meaning within a given frame of reference is compounded immensely.

So the science translator who aims at transduction to non-science does more than scratch his head in wonderment, asking himself whether "scientists really do speak the same language." Our third maxim comes to his mind:

(*C*) *Scientists do not speak the same language!*

It is not for me to say whether this maxim represents a good or a bad situation; each reader will have to form his own opinion. And it is not for me to indulge in an attempt at definitive moralizing, and to say that this represents deterioration or retrogradation of the language of science. If nothing else, it shows the limitations of the English language, the multi-functional role words play in science, and the necessity of invention to cope with a linguistic explosion for which our language was not designed.

It should be emphasized that the semantic equation approach is an excellent tool when properly used, that it enables the solution of otherwise very difficult problems in terminology, and that it will be employed to an increasing extent as time goes by.

For better or worse, it is far too late to change things radically. For better or worse, the little-languages of science will move away from those of non-science with increasing velocities—sciencese, by its very nature, is a "restless little-language." Like it or not, the translator deals with highly dynamic entities which are constantly in a state of flux and he must be constantly alert if he is to make the bridge between the examples given earlier.

What the foregoing boils down to is a fourth maxim:

(*D*) *The science translator and re-jargonizer must be both science philologist and non-science philologist if he is to make "non-existent science" real.*

Non-Science Jargon

At the other end of the spectrum are many and varied highly specialized non-science jargons. Of course, the desirable in-between "semantic bands" cover good, simple English but, as has been indicated, this does

[6] Fluorspar came before either fluorescence or fluorine, but both claim it as namesake. The prefix *fluoro-* is preferred for fluorescence, while *fluori-* would be more appropriate for fluorine. In his earlier writings on luminescence the writer probably didn't help matters much.

not always suffice and re-jargonizing must be relied upon. Many factors interfere with the use of the more desirable "linguistic wavelengths," particularly the highly efficient jamming resulting from the American public's reading and speaking habits.

In re-jargonizing into non-science media the leeway is greater, and the translator can often coin his words and expressions to suit the need (often with considerable impact). Thus: it frequently does little good to lean on a shop-worn expression like "a thermonuclear device carries 25 megatons TNT explosive power" unless it is propped up with something like "a 25 megawhammy wallop." This is what the average person can understand and remember.

Besides getting the message through, one of the end-values of non-science phrasing is that the memory of the sensor is not unduly taxed if he should later wish to re-communicate what he has absorbed. The hazard of course is that in re-jargonizing, oversimplification and conceptual marring can and do occur, and important parts of what is communicated are insulted or lost along the way.

A good example is the relatively new field of television which, although it has not received the attention it deserves, deserves utmost attention because of its great powers in molding human thought, attitude, and behavior. It is interesting to explore briefly one small part of television.

The television set and the typed actors and programs are referred to by various segments of the industry in many different ways, as expected in a medium where mercurial human elements are an integral part. They range from the most glowing of superlatives to the most derogatory of terms. Examples:

ETV (educational TV); vacuum-pack; lip-sinking (dubbing in English for a foreign language); nit-flicker (derogatory); Big Brother (paranoid connotation of being watched, not watching); bubble-board (derisive, from the numerous soap commercials); phosphor pie (connotating viewing and eating); cancer bucket, coffin seller, lung embalmer, cancer jockey, and others (all derogatory, from the many cigarette commercials and the alleged connection of smoking and neoplasia); UTV (universal TV, i.e., multi-lingual TV); TViewer; idiot-box (derogatory, some suspect with good reason).

Many strictly taboo words and expressions might be given, but these might make this magazine unmailable, so I will leave that problem to the philologists and etymologists who write scholarly works on such subjects. By way of note, it is suspected that nuclear, space, and military scientists and engineers are most adept at lacing their little-languages with colorful words and expressions.

Why Translate and Re-Jargonize?

There are many answers to this question. If there is a simple one, it is to get science over to the public. In turn, this often implies that "it's not always what you say, but how you say it." Maxim, (*D*) above, indicates still another answer: To make "nonexistent science" real. "To make people think" may be the ideal answer but, as most educators well know, this is not easy to do.

Properly translated, science does not lose dignity. Sciencese cannot regain dignity that was not there to begin with. Nor is the converse true: non-science lingo cannot lend a dignity when inherently it is unable to do so. Non-sciencese may or may not be offensive or irritating to the professional scientist but, in the absence of the more desirable simple English, it serves a common and valuable purpose.

Re-jargonizing is not to be confused with sensationalism. Perhaps such confusion does exist among scientists and is one reason why science writing is looked down upon. If a presentation is brief and right to the point—although partly in another language (seldom, if ever, is it wholly in another language)—it does not follow that the presentation is merely balloon juice, i.e., highly exaggerated ("balloon juice" is slang for helium used to inflate balloons). Accurate information expressed clearly and concisely should be the goal. This seems to be attained as often in non-science jargon as in much of the insipid sciencese of the professional.

It is well to remember that an *anti-science exacerbation* can occur whenever more is expected of science than it is inherently capable of. Re-jargonizing, or translating the "mystic language," therefore has an added unexpected value for science: it is one means of dispelling the widespread, vague, and sometimes uneasy suspicion with which science is frequently viewed by those who do not comprehend its methods and aims—and one way of helping to build and sustain rapport between scientists and the non-science public.

Moreover, science should protect itself from ill-founded abuse and misinterpretation. An editorial in *Science*[7] entitled "So Science says . . . " advocates the use of ridicule and protest to combat these abuses. When the scientist who doubles as translator couples his knowledge, experience, and prestige with appropriate ridicule the results may be most gratifying. It is up to him and his common sense to decide where ridicule leaves off and sarcasm, cynicism, carping, and ax-grinding begin. *Caution:* The line between effective ridicule and the ridiculous is indeed thin!

Like ridicule, humor deserves special care; it can be a powerful tonic or (like humor in politics) it can have unforeseen consequences. The translator who tries humor risks a posture which may be side-splitting to some and silly to others, so he must choose whether he wishes to risk conjoining humor with an apparently humorless science topic.

Those who contemplate the transduction of science will do well to follow the advice earlier quoted from THIS JOURNAL.[2] It is not a boilerplate proposition. It is, instead, one where the individual's efforts and occasionally his personality should get through to the reader. He must be able to use one simple word for six technical words—and still keep the meaning. This is not easy to do for one long in the habit of writing and speaking a particular little-language, and it will entail hard work at the outset and at times perhaps a false feeling of self-compromise.

Who knows how many young people will become our future scientists from having read such articles? This is the great obligation and responsibility of the science translator.

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[7] *Science*, **132**, 1749 (1960).

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Personalized Bibliography: An Approach to the History of Chemistry

There is a fundamental reason for studying the history of science in general and chemistry in particular; this is simply that we cannot properly understand any science without a knowledge of its history. As Dingle (*1*) has pointed out,

> The most distinguishing characteristic of science is that it is a progressive, age-long process of acquiring knowledge, in which each successive generation builds on the work of its predecessors and can accomplish nothing lasting if that work is not soundly based. In this it stands in striking contrast to every other human activity. In every form of art the work of each creator exists in its own right. The value of Wordsworth does not rest on that of Milton, nor Milton's on Shakespeare.

Lavoisier's chemical revolution, however, does rest on the earlier work of Priestley, Stahl, Boyle, and a host of other predecessors. In science and only in science does each successive worker start from the point which his predecessors had reached and provide a basis on which his successors will, in their turn, build. Upon consulting any paper in the *Journal of the American Chemical Society*, one will find at the end a list of references to earlier works which the author has consulted, used, and accepted as accurate. A study of any of the previous work cited by the author leads to references to still earlier work whose validity is assumed and which is necessary to the accuracy of its own conclusion; this process can be continued back at least to the 17th century from which time chemistry has been a gradually ascending structure. All that is done at any given time rests on what has previously been accomplished and leads to and supports what follows. This is, indeed, the nature of all science and a weakness at any point will imperil the whole structure above it. This will explain the revolution that occurred in chemical theory when Lavoisier demonstrated the falsity of the Phlogiston theory. For this reason it is clear that a study of the history of chemistry is necessary not only as a liberalizing influence but also for the successful understanding and prosecution of the science itself.

Contributions to our knowledge of the history of chemistry may be made in different ways. The first and most widely useful, but difficult to produce, is the general history. Synthetical work of this nature can only be prepared by the most competent historians and scholars like Partington, whose excellent "A Short History of Chemistry" (*2*) forms a good example. This type of endeavor is only achieved by judicious and liberal use of the other approaches we are about to consider—each of which is in itself a field requiring exact and careful research and necessitating special qualifications.

The second type of approach is the special history, such as White's "The Phlogiston Theory" (*3*). These deal with the study of a single period, theory, or development.

The third type is the biographical approach which will deal exclusively with the life and doings of an individual. For an illustration of this sort of endeavor I would cite More's "Life of Robert Boyle" (*4*).

A fourth category of approach is given by histories of individual industries such as Singer's monumental history of the alum industry (*28*).

A fifth class is provided by catalogues of individual chemical libraries, such as Ferguson's scholarly catalogue of the outstanding collection amassed by James Young (*5*). Such catalogues have considerable value—especially when relating to a subject such as the history of chemistry in which definitive bibliographical apparatus is lacking. However when consulting this type of compilation, an important limitation must be borne in mind and that is the fact that a collection will necessarily reflect the whims and fancies of the collector. In

Dexter Award Presentation

Denis I. Duveen (standing, left), president of the Duveen Soap Company, Long Island City, New York, is shown receiving a $500 check from Joseph B. Evans, executive vice president of Dexter Chemical Corporation, for his "noteworthy contributions to the advancement of the history of chemistry."

A plaque, part of the fifth annual Dexter Award, was presented to Mr. Duveen by Dr. Virgil Payne (far left), chairman of the ACS Division of History of Chemistry, which administers the Award. Second from left is Dr. Henry Guerlac, Cornell University, principal speaker at the divisional luncheon at the 138th Meeting of the ACS in New York, September 1960. Dr. Aaron Ihde of the University of Wisconsin, member of the Editorial Board of THIS JOURNAL, is on far right.

order to render service to those interested in the field I published such a catalogue of my own collection (*6*), and judging by the frequency with which it is quoted the compilation was needed and has been found useful. However, it is irritating to see books described, usually by dealers, as "Unknown to Duveen" or "Not in Duveen" in an attempt to make an item out to be a rarity when the truth probably is that the particular work did not figure in my library because I did not think it of sufficient interest for one reason or another. This is the big difference between a catalogue and a bibliography; the latter is definitive and complete—or at any rate it should be.

The Bibliographical Approach

This now leads up to the sixth method of approach and the one on which I wish to dilate at greatest length, namely, the bibliographical one. It now becomes necessary, therefore, to consider the meaning of the word "bibliography," which unfortunately seems to be greatly misunderstood. On the one hand, it is commonly but loosely used to describe incomplete and often relatively useless short lists of works; on the other hand, it is given an esoteric sense in which it is supposed to describe some recondite piece of scholarship which will be unintelligible to the uninitiated.

Dr. J. D. Cowley, late Director of the University of London School of Librarianship, used to maintain that there were three kinds of study which orthodox opinion would describe as bibliographical.

The separation of an author's *work* from his *book*, or the dress in which his work is eventually clothed, accounts for one kind of bibliography: the description of the book in such a way as to assist the textual or higher critic in the solution of his problem. The textual critic tries to determine the words most likely to have been used by the author and attempts to do this by eliminating errors made by copyists, typesetters, or proofreaders. The higher critic assumes that the existing text has been perfected and then asks whether the work under study is fully authentic. It is evident that a minute examination of the material form of a work must be of the greatest help in establishing the most authentic text; and that such an examination must lead to the consideration of the circumstances in which that form was assumed and the processes by which the work became a book. These circumstances and processes can be described; bibliographical description is in fact a description, reduced to a formula, of the method of publication of a particular book through the setting down of the relevant details of format, typography, arrangement of content, etc., etc. This kind of bibliography, then, is really the study of the material transmission of literary and other documents, and its ultimate aim is to solve the problems of origin, history, and text, so far as this can be achieved through minute examination of the material means of transmission. In this connection, Dr. Cowley always stressed his belief that the most important function of bibliography was to tell a story; but whereas the story of literature or the story of chemistry records the breaking forth of new creative impulses or new syntheses of knowledge and traces their origins and their influences, the bibliographical story is concerned only with the clothing in which any of these children of the human mind were first swaddled.

However, a second type of endeavor that may legitimately be called bibliography, according to Dr. Cowley's definition, arises from the fact that many of us are, so to speak, interested in clothing by itself—quite apart from the personality of the wearer. This study treats books as things in themselves and is mainly concerned not with their authenticity as documents, but with the method of their production. Through such work, the history of printing has come to be written from the recounting, by the description of successive examples, of the steps by which book production advanced.

The third kind of orthodox bibliography is concerned not with the description of the material means of transmission but with the analysis of the subject matter of books and is usually called subject bibliography. It is about this sort of bibliographic work in particular that I wish to speak and I prefer to call it enumerative bibliography. There are many types; the best known are in essence lists of books or articles about a specified subject or by a certain author, but there are many others such as lists of books published within a given period of time, books published in a given city or country, books published in a specific language, etc.

General bibliographies of chemistry are, as might be expected from their nature, few and far between. The most useful of such works is still Bolton's massive compilation (*7*) but the increase in the literature of the subject has become so immense as to render it virtually impossible for an up-to-date version of such a bibliography to be produced.

A number of good bibliographies of the works of individual chemists have been published and are proving of inestimable use to the scholar, student, and collector. Recent years have certainly seen an increase in the accuracy and comprehensiveness of such works, an excellent example of which is furnished by Mostrom's compilation relating to the works of Torbern Bergman (*8*). Other recent examples of such well-done and useful work are provided by Lindboom's bibliography of Boerhaave (*9*) and Holmberg's bibliography of Berzelius (*10*).

The Personalized Bibliography

However, such work can be rendered much more useful by expanding the annotations to include an account of the work being described in each of the author's written contributions, together with an explanation of its individual importance in the development of his theories and in the general scheme of progress. Such an expanded bibliography has come to be called a *personalized bibliography* or a *biobibliography* and its aims and methods have been succinctly stated by John Fulton:

> In recent years, largely through the influence of Geoffrey Keynes in England and a group of French bibliographers who flourished during the 19th century, the personalized bibliography has come to the fore, and with it has arisen a renewed interest in bibliography as a humanisitic discipline. If one wished to know fully about a man, the conventional biography usually offers insufficient detail; one must know when he began to write, have information about everything he wrote—published and unpublished—and what prompted him to undertake each individual work. This vital aspect of a man's life lies in the province of the bibliographer rather than in that of the biographer (*11*).

The famous medical bibliographer, Sir William

Osler, also expressed himself particularly well on the subject when he wrote:

> Not naturally dry, bibliography is too often made so by faulty treatment. What more arid than long lists of titles, as dreary as the genealogies of the Old Testament, or as the catalogue of the ships in Homer! What more fascinating, on the other hand, than the story of the book as part of the life of the man who wrote—the bio-bibliography....To be of value to the full-fed student of today a bibliography should be a "Catalogue Raisonne" with judicious remarks and explanations (*29*).

In England Keynes, although a distinguished and active surgeon, has found time to produce a number of most excellent personalized bibliographies of eminent scientific and other (*21*) figures; in this country John Fulton pioneered with his compilations of the works of Robert Boyle (*11*), Richard Lower (*12*), and John Mayou (*13*). A study of any of these bibliographies will indeed show that when all the published works of one man are assembled, listed, and physically described, with their content analyzed as to its origin, importance, and significance, the whole intellectual history of the subject unfolds with much more immediacy and clarity than in the best and most lucid biography which is, of necessity, based more or less on the external facts of that man's life.

I have described elsewhere how a concatenation of circumstances led me to an awareness of the fact that no adequate bibliography of the works of Lavoisier existed (*14*). At the same time came a realization of the surprising fact that the majority of his works were either virtually unknown or poorly understood. In particular, it was obviously little appreciated that Lavoisier was not only a great chemist, probably the greatest, but a noted administrator, economist, agriculturist, and sociologist as well—nor was his work in these other fields by any means superficial. The standard biography of Lavoisier was that of Grimaux (*15*) and this was only good in matters biographical: the bibliography contained in the work was grossly incomplete and inaccurate. Since this biography was written, all studies of Lavoisier's life and work with but few exceptions have drawn from this source and are necessarily little better than the original. Mention should be made of the contributions of Meldrum (*16*), McKie (*17*), Daumas (*18*), and Guerlac (*19*), which are noteworthy for their fresh approach but unfortunately are largely limited to certain phases of Lavoisier's endeavors and usually only those concerning chemistry. The need for a definitive study of Lavoisier was, therefore, apparent; Herbert Klickstein and I felt that a complete bibliography of his works would be the most useful step toward this objective. A personalized bibliographical approach was chosen, for by this means only, we felt, could the true stature of Lavoisier be demonstrated.

In our bibliography (*20*) we followed the principle of listing each memoir and every volume in which Lavoisier's name was given as one of the authors or which was issued by any organization during his tenure of executive office; we listed also those cases in which his authorship could not be incontestibly proved, even when the evidence suggested that he did not write a specific article although he signed his name to it. The circumstances were, of course, carefully pointed out in each individual case of this type. We not only described each item but analyzed each with regard to content and historical importance. The analytical commentaries were extensive but we felt that they would be especially useful because of the scarcity and consequent difficulty in consulting the originals of many of Lavoisier's works.

The compilation of this work was intended to prove a useful tool of reference for historians, collectors, and bibliographers alike, as well as the basis for a new evaluation of Lavoisier's greatness. It taught the compilers much and out of the necessary research work came detailed studies of such diverse special subjects as:

The Introduction of Lavoisier's Chemical Nomenclature into America (*22*)
Lavoisier's contributions to Medicine and Public Health (*23*)
Benjamin Franklin and Antoine Lavoisier (*24*)
Lavoisier and the French Revolution (*25*)
Medallic Portraiture of Antoine Lavoisier (*26*)
Lavoisier–The Innovator (*27*)

As far as chemistry is concerned, it would appear that Robert Boyle and Antoine Lavoisier are the only two figures to have formed the subjects of personalized bibliographies. The compilation of such studies inevitably leads to the discovery of new facts and a reevaluation of old ones. There are many eminent precursors who deserve to be studied in this way and a complete list would be very extensive; however, it is suggested that the following chemists should have personalized bibliographies of their works prepared:

Andreas Libavius	1540–1616
Johann Rudolf Glauber	1604–1670
Nicolas Lemery	1645–1715
Johann Joachim Becher	1635–1682
Georg Ernst Stahl	1660–1734
Carl Wilhelm Scheele	1742–1786
Joseph Priestley	1733–1804
Guyton de Morveau	1737–1816
Claude Louis Berthollet	1748–1822
Amadeo Avogadro	1776–1856
Johns Jacob Berzelius	1779–1848
Friedrich Woehler	1800–1882
Justus von Liebig	1803–1873
Jean Baptiste Dumas	1800–1884
Hermann Kolbe	1818–1884
Louis Pasteur	1822–1895
Marcellin Berthelot	1827–1907
Dmitri Ivanovitch Mendeleev	1834–1907
Emil Fischer	1852–1919
Marie Curie	1867–1934

This list is not intended to be in any sense complete but is only suggestive of some chemists of the last four centuries whose personalized bibliographies could fruitfully be compiled. Some in my selected list wrote and published comparatively little and therefore the bibliography would be relatively short; others published much. The field is wide open; the period to be studied and the amount of work involved can be chosen to suit the desires of the compiler.

I feel strongly that if only this limited program were to be followed much new information would be brought to light and a reevaluation of much chemical history could be made.

In concluding these remarks on the usefulness of bibliography as a tool for the historian of chemistry I cannot do better than quote Fulton again:

> A modern bibliographer must anatomize his books; he dissects

them with infinite patience, lifting their epidermis to find what lies beneath; he is concerned with their joints and ligaments, and has great delight in discovering parts which have been artificially replaced; he seeks for errors in the hand of the maker, but he views with charitable amusement all signs of human frailty. Bibliography is indeed an all absorbing occupation, but its devotee is frequently face to face with those who fail to understand the source of his enjoyment. A mere list of bibliographical idiosyncracies with mistaken signatures, pagination and gatherings, has little appeal to any one not a collector of books; and however much a bibliographer may pride himself on his "Purity" he has difficulty in justifying his existence if he fails to make his work useful to those not pursuing his specialized field. He must reveal something more than the mechanics of bookmaking. He can endeavor to assess the importance of a book; he may say how the author came to write it, or investigate the influence which it exerted upon his contemporaries (*11*).

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Ultra Low Conductivity Water

Applying an electrophoretic ion-exclusion technique, the National Bureau of Standards recently succeeded in preparing water of extremely low ion content. The water obtained has an electrical conductivity of 0.039×10^{-6} ohm^{-1} at 18° C, indicating a residual ion content which is equivalent to a sodium chloride concentration of one part per billion. Containing less than one third of the ionic impurities of the water prepared by Kohlrausch and Heydweiller in their historic purification experiments, this water approaches the theoretical conducitivity (0.0371×10^{-6} ohm^{-1}) more closely than any values previously reported in the literature.

The present research, undertaken by Wolfgang Haller and H. C. Duecker of the Bureau's staff, was part of a study of ion diffusion. In designing the purification procedure, two principal considerations were involved. First, instead of using multiple distillation as in the classic experiments, an electrophoretic procedure was applied. In this way purest water was obtained in 2 hours, while in the best previous study, 36 consecutive vacuum distillations were required. Second, the purification apparatus was designed to recirculate the purified water continuously through the electric field, thus immediately removing any ions which might originate from the walls of the apparatus and contaminate the already purified water.

By this kinetic method, it is possible to maintain the purity of the water if a container material with very low ion-release is used. While fused silica containers which had been exposed to water for ten years were utilized in former purifications, high-density polyethylene, platinum, and borosilicate glass which had been exposed to steam for a number of hours just before purification were found suitable for the present experiments. The steam condenses in the apparatus, thus rinsing its internal surfaces with a stream of freshly distilled water. At the temperature of this treatment, the diffusion of ions from the glass—which is the main potential source of ionic impurities—is many times faster than at the temperatures at which the apparatus is used, resulting in an effective depletion of the surface regions of the glass from objectionable ions.

National Bureau of Standards Summary Technical Report

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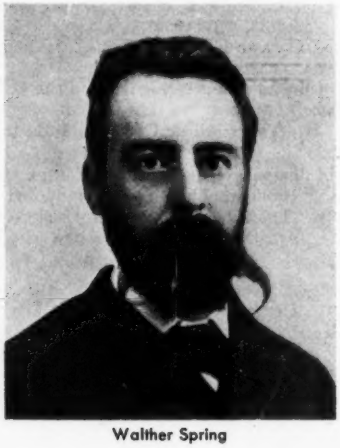
Walther Spring (1848–1911)

In the century which roughly preceded the present era, Belgium produced three chemists who acquired international fame, namely, Stas, Spring, and Swarts. Stas, a native of Louvain, was trained primarily at Paris under Dumas, who put his brilliant student on the path which he followed through most of his chemical career, the precise determination of atomic weights.[1] Spring and Swarts came later; the former was a student at Liege, the latter at Ghent, but they both went to Germany for their higher training.

Spring's father was of Bavarian extraction. He was on the medical faculty at Liege and later became Rector of that university. The son Walther held the chair



Walther Spring

of general chemistry at Liege for many years. He was a brilliant teacher and made a deep impression on his students, but he attracted but few disciples because he became a misanthrope at an early age and preferred to work alone in his laboratory. Because of his manual skill, he was able to carry out experiments which at that time were technically too difficult for most chemists.

Consequently, his scientific works were marked by great originality.[2] From among his most remarkable accomplishments, note should be taken first of all of his investigations on the behavior of solids subjected to extreme pressures of the order of thousands of atmospheres per square centimeter, pressures which at the end of the 19th century broke all records. In this way he showed that it is possible for metals to diffuse into each other if placed in intimate contact at ordinary temperatures and then pressed together. For example, copper and zinc form an intermediate region brass, a typical example of a solid solution. This was established at a time when van't Hoff had not yet popularized this concept. If analogous experiments are conducted with materials stemming from silicious rocks for instance, but without taking the precaution to remove the traces of air, humidity, or other impurities which separate the fragments being studied, a prolonged compression transforms the mass into an agglomerate of schist, whose leaflets are separated by thin layers of foreign elements.

Having thus discovered the mode of formation of certain typical rocks of the Ardennes region, Spring undertook a study of the waters of the Meuse River, which flows through the city of Liege. For more than a year he followed the flow day by day, noting the particularities which it presented. The water was sometimes green, sometimes blue, and sometimes brown, and this succession of colors led him to the question, what is the color of pure water? To arrive at the answer, it obviously became necessary to eliminate all traces of suspended matter.

To obtain optically "empty" water, Spring used two procedures. In the first he mixed equivalent amounts of ammonium hydroxide solution with a solution of aluminum chloride. The resulting colloidal hydrous alumina settled in time and carried down with it all of the suspended impurities. He assured himself of the optical purity of the water treated in this manner by passing a strong beam of light through the clarified water. Observed laterally, the beam was quite visible in the surrounding air because of the illumination of the suspended particles (Tyndall's effect) but it was completely invisible in the liquid. He arrived at the same result in his second procedure by attracting the electrically charged particles toward the poles of an electromagnet. Such optically empty water is practically colorless, but if it is observed through a layer ten meters long, it is found to be bluish. This finding was later confirmed by studying the absorption spectrum, a technique which was not yet in use at the time Spring carried out his investigation of this interesting problem.

He applied his clarification methods also to solutions of salts of the alkali and the alkaline earth metals. He found that the blue color remains intact; but if the salt hydrolyses, the solutions tend toward the green even though the salt itself is colorless. Finally, by means of this same technique Spring showed that the paraffin hydrocarbons are pale yellow and that the alcohols take on a progressively more greenish cast as the alkyl radical becomes longer.

[1] See TIMMERMANS, J., THIS JOURNAL, 15, 353 (1938) for the story of Stas and his accomplishments.

[2] His collected works were published in 1914–23 by the Belgian Chemical Society.

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Frederic Swarts (1866–1940)

Frederic Swarts devoted his entire professional career to the University of Ghent, where he was a student under his father who had been the collaborator with and eventually the successor to August Kekulé.[1]

Frederic Swarts exemplified tenacity of effort and work, having acquired the degrees of Docteur en Sciences and later Docteur en Médecine. He ascended one by one all of the steps in an academic and university career, and eventually became the Président du Comité National Belge de Chimie and Président du Comité Scientifique de l'Institut International de Chimie Solvay. He was competent in both organic and physical chemistry, and will always be renowned as the real creator of the chemistry of organic fluorine compounds. Before Swarts had entered this field, Beilstein described a mere dozen such compounds in his Handbuch; eventually Swarts added 40 more, including the parent "Freon" in 1907.

The preparation of fluorinated organic derivatives for many years had been acknowledged to be most difficult. Hydrofluoric acid does not attack the hydrocarbons; in addition it is difficult to handle because of its attack on glass. The use of elemental fluorine, which had been isolated in useful amounts by Moissan, required platinum equipment. Consequently, an indirect method had to be devised to overcome these difficulties. It was necessary to start with a chlorinated or iodinated derivative and then to introduce the fluorine by substitution. For this purpose Swarts prepared a wide variety of previously unknown compounds containing halogens, and used them as starting materials for the preparation of the corresponding fluorine compounds. At first he used a mixture of bromine and antimony trifluoride, later he employed antimony fluochloride, and finally mercury(I) fluoride. In aromatic compounds, the fluorination of a side chain goes more readily. Swarts isolated trifluotoluic acid which on oxidation yielded trifluoroacetic acid. Eventually he succeeded in preparing fluorobromo-chloro-methane (CHFBrCl), an asymmetric compound. Unfortunately, the antipodes racemize too rapidly to permit the isolation of the optically active forms.

[1] See GILLIS, J., THIS JOURNAL, **38**, 121 (1961).

Frederic Swarts

Being thus provided with a considerable stock of original materials, he began to study the most characteristic properties from the physical chemical standpoint. He showed that fluorine behaves like the other halogens but sometimes uniquely. For example, the acidifying function is much stronger in the fluorinated derivatives than in the corresponding chlorine or bromine organic compounds. With the aid of strychnine, he made the first resolution of a molecule containing two carbon atoms, namely, fluorochlorobromo-acetic acid. The C—F bond was found to be especially strong. Molecules containing it associate very little, have low surface tension, and very low boiling points. For example, trifluoroacetyl fluoride boils at −59°C, whereas acetyl fluoride boils at +20.5°C.

In 1919, Swarts began to study the heats of combustion of fluorinated compounds and verified the high heat of formation of such compounds. For example, the heat of formation of carbon tetrafluoride is 200 kcal/mol, double that of carbon dioxide.

The many years of dealing with these compounds had gradually affected his respiratory system, and at the close of his career he found it imperative to spend the winters in a warmer climate than that of Belgium. However, the occupation of Belgium by the Germans in the spring of 1940 prevented Swarts from taking his usual trip and he died during the following winter.

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A Summer Research Participation Program for High School Students

A large effort has been made in recent years by foundations, government, and the chemical profession itself to attract greater numbers of better qualified students into chemistry and change the deplorable stereotype of the research scientist into a more realistic image. Well known programs have been initiated to further and modernize the education of high school teachers and professors in small colleges, to increase subsidization (direct and indirect) of graduate research, and to provide science competitions among high school students. In the past three years the National Science Foundation has directly attacked the problem of recognizing and encouraging science interests in secondary school students of high ability by directly subsidizing efforts to bring these students onto college campuses on a part time basis.

Such a program in chemistry was held at the Polytechnic Institute of Brooklyn during the summer of 1960 and will be repeated in 1961: high school students worked on open ended laboratory problems (which could in most cases be classified as research) of a high level of sophistication. The sponsored program was patterned after an informal and successful Saturday program which had proceeded at the Institute for several years, since a considerable body of experience handling this age group in the laboratory already had been amassed. The number of applicants had increased yearly.

Introducing high school students to research is both new and interesting, as it ties together laboratory work, always a good pedagogical tool in an experimental science, and the excitement of problem solving. In the informal atmosphere of the laboratory, communication becomes easier and stereotypes fall victim to intimate personal contact. However, the practical problems of making the programs meaningful are manifold and difficult. In introducing research to high school students over the past four years we have solved some of these problems, have minimized others, and have spent considerable time thinking about those which remain unsolved. Following is a description of our summer program and an analysis of the effectiveness of its major features; we hope that others will find it useful in shaping their own programs.

Program Objectives

The educational goals were developed from the premise that the high-ability high school student needs to be led more deeply at each step through his formal educational career, rather than to be led faster. Therefore our goals were

(1) To encourage creative thinking about scientific experiment and theory by placing high ability students into an intellectually stimulating milieu;

(2) To enlarge the scientific interests of those already committed to a scientific career and encourage an interest in science among those who are undecided;

(3) To provide a realistic picture of the attitudes and attributes of the scientist and his work; and

(4) To impart specific factual information concerning the techniques and theories of the existing body of chemical knowledge in order to allow the student to proceed more rapidly through the early stage of his university education. He then may enrich his curriculum later with scientific subjects outside his discipline and advanced courses within it.

Participant Selection

To reduce 250 applicants to a reasonable figure from which the 30 participants could be selected was difficult, as students with scholastic averages less than 90% generally did not apply. Selection was finally accomplished using scholastic average (over-all averages and science and mathematics course averages were both requested), maturation (as indicated by extracurricular activities of an intellectual nature) and teacher recommendations. I.Q. test scores were requested but because several different tests are used in our area, the scores were used only as a minimum requirement.

All who were concerned with the program noticed major differences among the intellectual aptitudes of the selectees, differences too large to be explained by anything but a defect in our selection procedures. We feel that the difficulty lies with the differing standards of the secondary schools from which the young people come. The stiffer competition and more advanced level of instruction at a specialized high school such as Bronx High School of Science requires that a more absolute measure than scholastic average is necessary to identify the student for whom we are looking. The "profiles of standard scores" of the Iowa Test of Educational Development seem well suited to our selection needs as most students have taken the exam in our area by the 10th grade. The student's scholastic grades will then be used to indicate whether he is working to the extent required by his particular high school.

Laboratory

The nine week program was composed of three parts: laboratory, seminars, and lectures. Each student was assigned to a professor on our staff who in turn assigned the student a laboratory problem, laboratory space, and a graduate student to oversee his daily work. The student worked a 40-hour week, but punched no time clock and was free to leave when there was little to do and to stay late when his laboratory problem required extra attention. In this way the comparative freedom of laboratory work and the necessity of careful planning were emphasized.

During the first week the students became familiar with the library and were taught methods of searching the chemical literature so they could search out and read background material. Several times a week lectures were offered. Some concerned specialized subject matter in chemistry, some the techniques of chemical research, others the conceptual framework of scientific inference, and still others career opportunities in chemistry. The idea behind the lectures was neither to give the student specific information about a circumscribed body of knowledge (as in the ordinary high school or college course) nor to give the student a minimum amount of information designed to help his laboratory work, but to penetrate lightly into a broad spectrum of topics. The lectures were to interest rather than to inform. Learning enough about his laboratory problem to carry it out successfully was left to student initiative with the help of the professor and graduate student in the research laboratory. The laboratory work was specific and penetrating whereas the lectures were to give our students a broad insight into chemistry and the chemical profession.

The third part of our program was small weekly seminars, of eight students each, in which they orally presented the results of the week's work. The seminars were run by two outstanding high school teachers from New York City schools who spent one day a week with us running the seminars and talking individually to the students. The seminars were to encourage all students to formulate their ideas of their work at intervals and to broaden each participant's experience by allowing him to learn of the work of others outside his laboratory. These seminars could also be utilized by the director to exert greater control over the participants by giving him a weekly account of each student's progress.

The nine week length of the program allowed the maximum of laboratory work without encroaching upon the public schools, and left the student a two week vacation at the end. It was decided that the maximum possible time would be necessary because original experimentation is time-consuming and often time-wasting. The time wasted in following blind alleys and correcting blunders, however, is not *lost* time for the high school students and in fact can be quite educational. We found that nine weeks was adequate time for outstanding students to learn the necessary laboratory techniques, to understand the scientific basis of the problem and realize where in the framework of science the problem fits, and therefore to accomplish limited laboratory results.

Some high school students worked along with a graduate student on the latter's thesis problem; others worked out problems related in a general way to work proceeding in the laboratory. As long as the graduate student does not use the student as a "pair of hands," the two methods seem equally efficacious in producing laboratory results and accomplishing our stated objectives. The students, however, overwhelmingly preferred to work alone on their own problem, though none of them came forward at the beginning of the summer with a worth-while project of his own devising. Our experience shows that a two-week introduction to the techniques of research preliminary to beginning the problem is very advantageous. Students who learned the techniques as the problem progressed during the nine weeks generally did a less satisfactory job. They accomplished less and the quality of their work was inferior.

An analysis of the 1000-word final report required of each participant gives an indication as to the most successful type of problem for research participation programs. We find that problems with limited conceptual difficulties are much preferable even when the problem involves considerable manipulative skill. The latter can be easily taught, but it should be remembered that manipulative skill is important only as it helps the students carry out the larger objectives of the program. Thus, it is possible to err in the direction of making the problem too simple and too repetitive. Our experience suggests that it is better to set these very bright students too difficult a problem than too easy. Several examples from our summer program are given below to support these conclusions:

One girl synthesized carbobenzoxyglycine-*p*-nitrophenyl ester from carbobenzoxyglycine to acquaint herself with the basic techniques of synthetic organic chemistry. She then prepared the *p*-methyl ester of carbobenzoxyaspartic acid from l-aspartic acid and carbobenzoxy chloride. Both synthetic procedures were previously untried. The student grasped the basic structures involved and the functional group changes but found the infrared spectral and optical rotatory measurements difficult to interpret, with her limited knowledge.

A boy purified a sample of vinyl acetate by distillation, polymerized it by a free radical technique, and then precipitated a relatively homogeneous (in molecular weight) fraction. He measured the viscosity of the sample and from a relationship between viscosity and molecular weight he determined the average molecular weight of his preparation. The work went smoothly and the student appeared to grasp well why viscosity was a measure of molecular weight. The quantitative relation between these two variables, however, was beyond his ability.

Another boy studied the rate of the following aqueous reaction spectrophotometrically:

$$MnO_4^- + 8H^+ + 5Cl^- \rightarrow Mn^{2+} + 4H_2O + {}^5/_2\,Cl_2$$

and determined the concentration dependence of each of the reactants. A large amount of data was collected which will eventually be published. Order and molecularity escaped the student completely, but the less abstract idea of mechanism seemed clear.

A second girl measured the effectiveness of various drying procedures on transition metal chlorides using the disappearance of the 3μ water band in the infrared spectra. She complained at the program's end that the problem was too simple and repetitive.

In each case the student used considerable advanced equipment, did a number of simple laboratory operations (filtration, mp, titrations), and learned in detail about a specific area of chemistry.

Where the first weeks of the students' time were devoted to instruction in laboratory techniques, the students were uniformly more successful. The first student below spent two weeks preparing acetanilid, luminol, and methyl-β-naphthyl ether, all introductory preparations in our organic chemistry laboratory.

The first student prepared pyrazinoyl hydraside starting from pyrazinoic acid—a several step synthesis which involved a high pressure hydrogenation.

A second student spent three weeks preparing acetanilide, acetylsalicylic acid, phenolphthalein, fluorescein, and crystal violet. During this period he also fractionated an ethanol aniline mixture and chromatographed a vegetable pigment and an indicator dye. He then prepared a series of hydroxamic acids including benzohydroxamic acid, salicylhydroxamic acid, and pyrazinylhydroxamic acid.

A third student spent two weeks synthesizing $CO(NH_3)_6Cl_3$ and $Na_2Fe(CN)_5NO{\cdot}2H_2O$ and then spent the remaining time isolating $AuCl_3{\cdot}PCl_3$ and analyzing it.

Although the above examples indicate that synthesis work makes the best type of student problem (and admittedly one most easily finds a suitable problem there), it is possible to find successful non-synthetic problems as the following three examples show:

One boy studied the uptake of polymethylmethacrylate from solution by iron powder using a variety of solvents. His report included the absorption isotherms.

A second student used a colorimetric method for the determination of chromium in the dichromate form and then checked the accuracy of his method by analyzing some steel and ore samples.

Another built a cell for the determination of the Raman spectrum of dimethylacetylene. The cell was later used by a graduate student after the Summer Institute ended.

The selection of laboratory problems for this age group is not an easy task and the students were not all equally successful in carrying out the assigned work. Analysis showed that the students who performed least successfully did so for three reasons: (1) poorly designed problem, (2) inadequate direction, and (3) poor motivation. Of the three students who did noticeably worse than the group average, two did so because of reasons (1) and (2). The third student, whose qualifications looked good at selection time, showed a decided lack of interest. These cases point up one of the pitfalls of such a program as this wherein each student is allowed to progress at his own speed.

Seminars and Lectures

The seminars fulfilled their purpose admirably, *after* the students had participated in several. The reason for the unsatisfactory beginning is twofold: it took students two or three weeks to get a comprehensive grasp of their problems; they initially treated the seminar as a lecture until we strongly pointed out that criticisms and questions were expected. At the end of the nine weeks the seminars were most informal and the comments and criticisms were quite pertinent and knowledgeable.

The lectures, on the other hand, were the least desirable feature of the program. The director pointed out to each lecturer that the students were very bright and quick and only lacking in fundamentals, so that if the lecturer filled in the fundamentals, the subject could be carried to a meaningful depth. Our idea of student quickness however was exaggerated and many of the lectures on chemical principles simply passed over the heads of many students, leaving them confused. The lack of connection between talks appeared to make them less interesting to the student. It appears that their meager previous scientific knowledge did not allow them to see the unifying threads weaving the lectures together and, bright as they were, they could not assimilate the background material rapidly enough to keep pace with the speaker.

Despite this difficulty, we feel that a lecture series is a good thing as it brings the students together at regular intervals, imparts general chemical information and helps fill the inevitable waiting periods in experimental work. A short modern course in chemical principles and practice will be given next year, supplemented by lectures on the scientific method and the chemical profession.

Staff and Facilities

Most important to the success of our program were interested faculty members and graduate students; almost as important was the active graduate research program which functions in the summer months as well as during the year. Although most of our participants had already taken high school chemistry, the laboratory portion of our program had little relation to their high school laboratory experience. Consequently, an enormous amount of time had to be devoted to patient instruction of these young people in basic laboratory techniques. A professor's time is both limited and valuable and, in the average case, little more of the professor's time could be made available to the high school student than is normally devoted to a B.S. thesis student. Thus, the hour-by-hour direction fell to the graduate students working in the same laboratory. Pedagogically speaking, if the graduate student has the proper spirit, this is an excellent arrangement as the two students are not too far distant in age and experience. We were apprehensive initially, however, about the already overburdened graduate student's reaction to extra responsibility.

We have found that this arrangement works excellently in practice and any success that our program had can be attributed largely to the excellent working relationship which developed between these two groups of students. By interviewing the graduate students informally, it was found that many liked the position of "research director"; others felt that the high school students helped rather than hindered their thesis work. Since we have a large number of graduate students at the Polytechnic Institute, we were able to utilize mainly those students who participated out of free choice.

A sizable ratio of graduate students and staff to high school students is important in a laboratory participation program since each student is given a great deal of individual freedom. When staff members are always close by in the laboratory, waiting times can be fully utilized. In these periods the students can help a graduate student on his project, filter a solution, take a melting point, etc., or go to the library. Even if only a "bull session" results, scientific information and attitudes are likely to be communicated.

The drain of this summer program on the department's resources was considerably greater than we had anticipated from our experience with the informal Saturday program mentioned earlier. It diverted a portion of our staff and graduate students from their personal research, used considerable laboratory space and glassware, and slowed services such as infrared analyses. In our minds a first class program must, by its nature, indent itself markedly into the normal departmental operation; and the best one can do is to minimize the effect by careful choice of problems. An obvious and important way of accomplishing this is to choose problems which require a minimum of complex equipment—a good idea in any case. In practice this is not as easily done as one might think, as the average faculty member is accustomed to heavily instrumented modern laboratory procedures.

Program Cost

There was no cost to the student nor was he reimbursed for any expenses other than half his luncheon and commuting cost. The fact that each student attending missed the opportunity to earn money from a summer job evidently discouraged students from the lower income class; an informal poll of our students showed them all to be from middle and upper income families.

The cost of chemicals, analyses, and glassware came to $1950, a rather considerable sum—and $1700 of the total was for glassware. Assuming that only a small part of the glassware found its way into student home laboratories, this represents a substantially greater breakage rate than among our advanced undergraduates. The real total was something over the $1950 figure quoted above because only chemicals checked out of the stockroom were included.

Staff and Student Opinion

The director and those responsible for the over-all operation of the Summer Institute felt that for the majority (90%) of students whose problems had been well chosen and who had been given patient aid and direction in the laboratory, the program was an unqualified success and the students had an informative and stimulating summer. No examinations were given; our opinion is based upon the student reports and upon talking personally to both high school and graduate students. The majority of the students did a considerable piece of laboratory work of an open ended nature and *knew* what it was that they had done. Enthusiasm among the students for the laboratory work was immense, so great in fact that they resented the seminars and lectures as intrusions into their work days. The reaction among graduate students to having the high school students working among them was uniformly good. There appeared very little antagonism between the two groups and an unexpected spirit of comradeship was built up and maintained. The reaction among the professorial staff was more mixed. Some felt the end result would not be worth the considerable expenditure of effort but others are eager to assist next year.

High school student opinion was polled in the last seminar and what follows is a concensus of their remarks: The students were unqualifiedly in favor of the research participation idea and felt that a lecture course of study or working on a set of advanced laboratory experiments would not have interested them nearly as much nor made learning as painless a process. They cared not at all for the lectures, feeling that they were too disconnected and in many cases above their heads; they liked the idea of having a few lectures at the end of the day's work, but could not arrive at a set of topics which would be of interest to all. They felt the time period was too short (the program staff feels this is mainly due to the type of work they did and not, in most cases, to an overambitious choice of problem). Finally, they uniformly agreed that they had a much better picture of the research scientist and his work. Over half the students applied to continue their work in our Saturday during-the-year program.

Backgrounds of Graduate School Chemistry Faculties

Possible changes or realignments in the undergraduate chemistry curriculum are being discussed at all levels from the ACS Committee on Professional Training down to individual departmental staffs. Many factors are involved in determining the course of these discussions. One is almost sure to be the distribution of academic chemists according to field of training, since discussions of the "importance" of a field unfortunately sometimes involve loyalties bordering on emotional reaction.

It may help to promote a balance in such discussions if some figures are known.

To get a statistical picture of this problem, we analyzed the data in the "Directory of Graduate Research" (1959), prepared by the Committee on Professional Training of the ACS. The data are from 122 schools which offer the Ph. D. degree in chemistry. These data do not include the separate departments of biochemistry or chemical engineering, but some do contain biochemistry and chemical engineering as a part of the chemistry department.

The following table collates information on the fields listed by faculty members of the 122 institutions. The classification "other" includes those listed as biochemistry and chemical engineering and those making no designation.

Total number on 122 chemistry faculties	Number in				
	Organic	Physical	Inorganic	Analytical	Others
2076	625 (30.1%)	700 (33.7%)	247 (11.9%)	223 (10.7%)	281 (13.5%)

In addition, of the 122 schools listed, 27 had no inorganic chemist listed as such on the entire staff (22.9% of the schools) and 29 schools had not one single analytical chemist listed as such (23.8% of the schools).

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Educational Activities of the Western Connecticut ACS Section

Almost all the local sections of the ACS have Education Committees, and indeed the Washington office has an Educational Secretary. These efforts are undertaken in response to a national awareness of a need for more and improved science education. The question arises, however, how can these groups best serve their communities and their Society? This report describes the endeavors of the Western Connecticut Section.

The structure and functions of this committee have developed over the past decade from a small subcommittee of this Section's Public Relations Committee to the present widespread division of labor.[2] It is felt that the involvement of a larger number of the members of the section has several advantages: the workload is shared, and the sense of personal interest is maintained. The Committee is reconstituted each year to provide flexibility, but interested members usually continue to serve in various capacities.

The goals of the Committee[3] have been confirmed by each organizational meeting. These are to foster a more scientifically informed public; and to contribute to a more general interest in science in the school systems of the area, at the elementary school, secondary school, and college levels. These goals have been attempted by working with and through the students, teachers, professors, administrators, and other interested agencies (including other professional societies and teacher-"layman" groups). The method has been to use eleven "functional" subcommittees. Since the total Education Committee has 40 members, many of these serve on more than one subcommittee. The functional subcommittees serve as follows:

Awards and Scholarship. The Awards Program (started in 1952) and the Scholarship Grant (started in 1958) include presentation of a scroll and a one-year subscription to *Scientific American* to the outstanding chemistry student in each of the Fairfield County secondary schools participating.

Dues of the members of the Western Connecticut Section support the Scholarship. Each of the students selected by his school for our Excellence in Chemistry Award is eligible to participate in competition for our scholarship of $250. The schools are requested to name their choices for the award by any method they choose. The scholarship is given to the student making the best score on a written examination, usually designed to last three hours. Sealed copies are sent to the schools participating. Under the supervision of a school official, the student works on the examination for the allotted time. The examination is sealed into another envelope and returned to the Committee for grading. In 1959–60, a three part examination was used. Part 1 involved a series of multiple choice questions, part 2 had objective questions where more detailed answers were necessary; and the final part called for an essay of 500–700 words on a subject announced at the time of the examination.

The chemistry teacher of each scholarship winner is presented with a one-year membership in the American Chemical Society. In addition, usually, two teachers are sponsored to attend the NEACT Summer Conference.

High School Science and Mathematics Journal. A complete description of the first edition of this journal appeared in *C & E News.*[4] It is expected that future papers for the journal will come from students participating in our science seminars and in science fairs. Additional nominations will be solicited from school science faculties. The first issue was published with the financial assistance of the American Cyanamid Company. As an extension of this program, consideration is being given to the possibility of sponsoring a type of Meeting-in-Miniature, where the papers would be presented.

Information Service. The committee is compiling a file of the specialties of many of our 900 local ACS members. The schools have been informed of the file and have been instructed in its use. If a teacher has a specific question (either of his own or from a student) on chemistry or science in general, or if the teacher has a given topic to be discussed with an expert, the Information Committee will attempt to provide the qualified man. Under consideration is a proposal to provide each school with self-addressed post cards or inquiry forms which could be submitted as soon as a question or problem arose.

A major difficulty which still must be resolved is that of liability in furnishing information. Previous correspondence with the National Headquarters of the ACS and with the Attorney General of the State of Connecticut has still left the answer to the liability question much in doubt. Until it can be completely established that this committee will not be held liable for information which might be incorrectly used, the Information File must be handled with considerable discretion. This definitely is a serious limitation.

Presented in part before the Division of Chemical Education at the 137th National Meeting of the American Chemical Society at Cleveland, Ohio, April, 1960.

[1] Chairman, Education Committee, Western Connecticut Section, ACS, 1960 and 1961.

[2] RUSSELL, H. R., J. CHEM. EDUC., **33**, 592 (1956).

[3] *Chem. and Eng. News*, **36**, 76 (November 24, 1958).

[4] *Chem. and Eng. News*, **38**, 66, 69 (August 1, 1960).

Liaison with Metropolitan Bridgeport Science Teachers Association. A lecture-demonstration program was organized and presented before this group. Our major effort involves contributions to the Science Fair held in this area, supplying judges for the fair and awarding prizes for the outstanding chemistry exhibit in the Senior Division and the outstanding physical science exhibit in the Junior Division.

Liaison with Science Education Association of Western Connecticut. In 1958–59, a program was presented at a meeting of the Association. It consisted of four demonstrations—corrosion, photochemistry, chromatographic separation, and paper making—with instructions and materials to enable the teachers to repeat them in their classrooms.

Literature Service. Career literature is made available to the schools. "Shall I Study Chemistry?" (published by the ACS) has been distributed to all the area high schools. Packets of career literature and a bibliography of career and guidance literature are made available and are distributed to the schools. Old scientific journals are collected from ACS members and are sent to interested schools. Some attempt is made to suggest films which might be pertinent.

Publicity. The efforts of this subcommittee are complementary to those of the Section's Publicity Committee and the Public Relations efforts of the various industries in the area.

Science Seminars. During 1958–59, one six-session seminar series was held in Darien and another in Bridgeport, with about 35 exceptional students selected for each. The informal sessions were held on Saturdays and were well attended and enthusiastically received. In 1959–60, in cooperation with the two science teachers associations, a seminar series again was sponsored for a selected group of secondary school students. The program consisted of science lecture-discussion periods conducted by outstanding professional men. Its purpose was to give students an opportunity not ordinarily available to hear and question scientists who are specialists and experts in their particular fields. Sessions were held on alternate Saturdays at three locations, Fairfield, Darien, and Wilton, Connecticut, from 9:30 to 11:30 A.M. Attendance was about 35 in each group and was limited to students who had demonstrated outstanding scholarship and either had completed three years of high school mathematics or were enrolled in their third year. The 1960–61 sessions are being held in two locations. A Fall series of four lectures and a Spring series of six to eight lectures were arranged with lectures running on consecutive Saturdays and an 11-week interval between Thanksgiving and the start of the Spring series in February.

Application forms for the seminar were sent with a description of the program to the head of the science department of each secondary school in Fairfield County, to be distributed to students who were interested and qualified. As might be expected, this technique for selection depended on the assistance of the school faculties in evaluating and recommending candidates. The program has been exceptionally well received by faculties, students, and discussion leaders (seminar speakers), and will be continued in 1961–62 with perhaps some expansion.

Speaker's Service. An outgrowth of the Information File, an educational speaker's service was established in 1960. This service works in conjunction with the Section's regular Speaker's Bureau, but specializes in presentations to the schools (grades 3–12).

A list of topics with a brief summary of each one was sent to all school superintendents, principals, and science coordinators in this area. Response to this specific listing was extremely good; it appears that specific offers are more readily accepted than generalities.

Science and Arts Camps Inc. Program. A further extension of the Speakers Service was initiated in October of 1960. A Science and Arts Camp for "talented" students in grades 4–6 had been inaugurated during the summer of 1960. The organizers desired to have a year round continuing program and asked that we consider a series of Saturday morning lectures for the campers. A 20-week program of 40 lecture-demonstrations was arranged and is currently in progress. The children are split into four groups of 30–40 each, and each group comes to the Laboratories of American Cyanamid Company on five consecutive Saturdays. At the Laboratory, the children are divided into two sections and each section discusses a topic for one hour. The sections exchange places and a new topic is presented for the second hour.

Facility Survey. Many schools have inadequate or non-existent laboratory facilities, and many school boards and citizens are not aware of this situation. Certain general approaches to the problem are: do not interfere with school systems at all; make a survey of science facilities and publish the results, if possible; work with individual school systems and teachers directly to criticize and to improve science teaching and facilities; set up an accrediting system.

In this area the committee would welcome suggestions on how to approach the problem, on the true criteria of adequate facilities, and especially advice on presenting the results without undue antagonism and with substantial hopes for improvements.

Exhibits. More specific efforts to assist with the problem of facilities included the preparation of exhibits where the student could actually handle the materials to gain an understanding of principles involved. One such exhibit was a colorimeter donated by Dr. G. B. Levy of Photovolt Corp. The colorimeter could be assembled and then put to experimental use.

Initiating the Programs. At the beginning of each school year in September, the chairman of the Educational Committee sends a letter to the individual superintendents of school systems, principals of senior and junior high schools, science coordinators, and science teachers. This letter outlines the areas of interest and functions of the various subcommittees which have been described in this article. Particular attention is called to the speakers available, the awards, the seminars, and the journal. Subcommittee chairmen are listed with addresses and business and home telephones.

Comments invited. While these multitude of activities are under way and an offer is made to provide additional services if necessary, we feel that we do not receive as many requests as our committee members have indicated willingness to handle. Is this to be expected from such a program? Have other such committees had these difficulties? Are there suggestions as to means to overcome the restraint in asking for help?

Chem Ed

BOOK REVIEWS

The Book of the Atom

Leonard de Vries, translated by *Eric G. Breeze*. The Macmillan Co., New York, 1960. viii + 267 pp. Illustrated. 15.5 × 22 cm. $3.95.

This delightful little volume, written primarily for young people of perhaps high school age, traces many of the significant developments in atomic science from the time of the Greeks down to the era of fission, fusion, and nuclear reactors. While it is intended for the younger generation, many a lay-adult will find it very interesting reading, informative and, more important perhaps, scientifically accurate. Here is an unusual combination of creative writing interwoven with abundant anecdote and historical background material, and built on a framework of sound factual information—perhaps a style which would assist greatly in making scientific books more generally attractive and salable.

The book is a translation of a work originally published in Holland under the title "Het Atoom." The Dutch flavor is refreshingly different, and we need reminders that Holland, too, is making its contributions toward "Atoms for Peace."

While there is scarcely a mathematical equation in the entire book, the author has nevertheless given attention to establishing in the mind of the reader quantitative concepts such as comparative sizes of atoms and nuclei, relative masses of atoms and nuclear particles, comparative energy releases in different types of nuclear processes, etc.

The author seems to have a good sense of holding the interest of his audience. As the story progresses into the more modern nuclear work there is greater use of anecdote to sustain the attention span in what might easily become some of the duller spots. The stories of the first chain reaction in the atomic pile constructed under the stands at Stagg field in Chicago and the first atomic bomb test in New Mexico were a bit tedious to the reviewer perhaps because of numerous exposures. On the other hand, the elements of drama and suspense with which they were written might prove appealing to a young person hearing them for the first time.

The first quarter of the text traces the history of atomic concepts down to the era of modern physics (about 1895). The remainder of the book deals with radioactivity, atomic structure, and nuclear phenomena. Interspersed here and there are chapters on such topics as fuels, sources of uranium, etc.

The volume closes appropriately with a few words of challenge to young people: "Therefore prepare yourself now for your work in the world of tomorrow, for it will be your world." Accurate, yet imaginative, writing such as this should do much to kindle the interest of young people in the sciences.

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Reviewed in this Issue

Leonard de Vries, The Book of the Atom
H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow and *P. D. Zemany*, X-Ray Absorption and Emission in Analytical Chemistry
J. R. Partington, A History of Greek Fire and Gunpowder
Arthur C. Cope, Editor, Organic Reactions. Volume 11
William Horwitz, Editor, Official Methods of Analysis of the Association of Official Agricultural Chemists
J. W. Linnett, Wave Mechanics and Valency
Mary Belle Allen, Editor, Comparative Biochemistry of Photoreactive Systems
Arnold Weissberger, Editor, Physical Methods of Organic Chemistry. Part 3
Arnold Weissberger, Editor, Physical Methods of Organic Chemistry. Part 4
W. D. Kingery, Introduction to Ceramics
Harry C. Gatos, Editor, The Surface Chemistry of Metals and Semiconductors
O. A. Battista, Mental Drugs: Chemistry's Challenge to Psychotherapy
T. Bassiri, Introduction a l'Etude des Parfums
Eugene G. Rochow, Editor, Inorganic Syntheses. Volume 6
Harry H. Sisler, Calvin A. VanderWerf and *Arthur W. Davidson*, College Chemistry: A Systematic Approach

X-Ray Absorption and Emission in Analytical Chemistry

H. A. Liebhafsky, H. G. Pfeiffer, E. H. Winslow, and *P. D. Zemany*, all of General Electric Research Laboratory, Schenectady, New York. John Wiley & Sons, Inc., New York, 1960. x + 357 pp. Figs. and tables. 16 × 23 cm. $13.50.

It is indeed rare to find a book written on such a technical subject as "X-Ray Absorption and Emission" that is superb. This scholarly presentation is clearly written and adequate in every respect. The reviewer is not in complete agreement with the statement that "the chemistry is going out of analytical chemistry" yet realizes that every analytical chemist must keep abreast of the theory and application of the many instrumental techniques. These authors have produced in book form a wealth of material for such a chemist.

The 10 chapters include "Origin and Properties of X-rays," "Absorptiometry with Polychromatic Beams," "X-ray Spectra and X-ray Optics," "X-ray Emission Spectrography," "Notes on Equipment," "Representative Spectrographs," and "Reliability of X-ray Emission Spectrography." Throughout the material good and consistent nomenclature is used. At the end of the first chapter an extensive summary of "Literature on X-Rays" is given. The many figures and tables are complete and clear. The figures for which photography has been employed are unusually good. Many references to the original literature are given. Extensive cross references are indicated by the inclusion throughout of section numbers in parentheses.

The text is supplemented by seven appendixes including tables of "The Approximate Intensities of X-ray Lines," "Wavelengths of Characteristic X-ray Lines," "Relation of Wavelength to Diffraction Angle for Common Monochromators," and a "Bibliography of Element Determinations." Good author and subject indexes are included.

The reviewer found no errors or faults with the book. The text is technical, but easily read, and is written "to be useful even if read only in part." It is to be highly recommended to any physicist or chemist interested in such material.

J. M. PAPPENHAGEN
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Gambier, Ohio

A History of Greek Fire and Gunpowder

J. R. Partington, University of London, England. W. Heffer & Sons, Ltd., Cambridge, England, 1960. xvi + 381 pp. Figures. 19 × 25.6 cm. 70s net ($9.40).

This study by the eminent English historian of chemistry examines the use of chemical incendiaries and explosives from ancient times up to the perfection of black gunpowder production during the Chemical Revolution. As Lieutenant-General Sir Frederick Morgan points out

in the Forward, besides being a history, the book is an obituary since it is published at a moment when nuclear explosives appear to have sounded the death knell of "traditional" chemical explosives.

Professor Partington points out that the use of incendiary materials was commonplace even in ancient times. This is brought out from his extensive examination of manuscripts and evaluation of scholarly studies. His handling of source materials leads to convincing conclusions regarding some of the knotty problems and legends prevalent with regard to the subject. Very frequently he draws directly from Greek, Latin, German, and French texts to demonstrate his points, or on occasion, to show the reader that a certain conclusion is not possible.

A lengthy chapter deals with the use of incendiary materials in warfare, particularly with Greek fire. With respect to the chemical nature of the latter, the book fails to arrive at a definitive answer, but examines the various incendiaries used before the development of gunpowder and rules out five of them as not satisfying the necessary properties. The sixth he suggests as most probably the substance which figured so prominently in the defense of Constantinople against the Muslims and the defense of Palestine against the Crusaders—a liquid composed of a petroleum distillate thickened with resinous substances, and possibly with added sulfur. Saltpeter (KNO_3) as a possible component is ruled out because of lack of knowledge of the pure salt before the thirteenth century.

The introduction of gunpowder into warfare followed the development of saltpeter production. The confusion of saltpeter with natron is examined, with the conclusion that the term, as originally used, referred to sodium carbonate. Although not directly related to the subject of the book, there is a good treatment of the production and use of natron and potash in early times. A long chapter on saltpeter relates the known history of the salt and includes a comprehensive examination of the written statements about its production up to the beginning of the nineteenth century.

The author points out that gunpowder was known to Roger Bacon from Arabic sources, dismissing the legend of its discovery by the Franciscan friar. He likewise dismisses Berthold Schwartz and the invention of firearms as unfounded legend, particularly since no evidence for the cleric of that name exists. The use of cannon early in the fourteenth century is indicated in the frontispiece reproduced from an illustration from a manuscript dated 1326. Numerous descriptions of early cannon are included. Separate chapters deal with gunpowder in Muslim, Indian, and Chinese records.

The book is a valuable study of a complicated subject. It fills a place in the history of chemical industry alongside the author's definitive work on the origins of applied chemistry.

AARON J. IHDE
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Organic Reactions. Volume II

Edited by *Arthur C. Cope*, Massachusetts Institute of Technology, Cambridge. John Wiley & Sons, Inc., New York, 1960. vii + 501 pp. Figs. and tables. 16 × 23.5 cm. $12.

This is the eleventh volume of the well-known series dealing comprehensively with selected organic reactions. Published under a new Editor-in-Chief, this volume maintains the familiar format and high standards of earlier members of the series.

The chapters included in this volume are: The Beckmann Rearrangement, by L. G. Donaruma and W. Z. Heldt; 156 pp. 596 ref. The Demjanov, and Tiffeneau-Demjanov Ring Expansions, by P. A. S. Smith and D. R. Baer; 32 pp., 111 ref. Arylation of Unsaturated Compounds by Diazonium Salts (The Meerwein Arylation Reaction), by C. S. Rondestvedt, Jr.; 72 pp., 161 ref. The Favorskii Rearrangement of Haloketones, by A. S. Kende; 56 pp., 130 ref. Olefins from Amines: The Hofmann Elimination Reaction and Amine Oxide Pyrolysis, by A. C. Cope and E. R. Trumbull; 177 pp., 398 ref.

All of the reactions discussed are timely and interesting; the reviews concerning the Beckmann rearrangement and the preparation of olefins from amines are especially welcome. The pyrolysis of amine oxides also has been discussed in a recent issue of *Chemical Reviews;* however, the two surveys are complementary. To date, no reactions have been reviewed in more than one volume of this series, although 20 years have now elapsed since Volume 1 was prepared. It is hoped that future volumes will contain a second treatment of some of the more important organic reactions along with reviews of reactions not discussed previously.

ROBERT K. INGHAM
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Official Methods of Analysis of the Association of Official Agricultural Chemists

Edited by *William Horwitz*, Association of Official Agricultural Chemists, Inc. 9th edition. Association of Official Agricultural Chemists, Washington, D. C., 1960. xx + 832 pp. Figs. and tables. 18 × 27 cm. $17.50.

This is a book of methods for analysis of foods, feeds, fertilizers, drugs, pesticides, and other materials related to agricultural products. One may find here, for example, procedures for determining phosphorus in fertilizer, DDT in dusting powder, phenol coefficient of disinfectants, alcohol in distilled liquors, vitamins in enriched bread, fat in milk, vanillin in vanilla extract, protein in stock feed, codeine in tablets, or glycerol in vanishing cream. Methods are adopted by the Association only after tests at several different laboratories, all following the proposed method on the same sample, agree on results of analysis. These planned collaborative studies insure that the official methods are reliable, have practical application, and give reproducible results in the hands of professional analytical chemists.

The Association of Official Agricultural Chemists, familiarly known as the AOAC, was organized in 1884 by State and Federal Chemists who were in charge of enforcement of state fertilizer laws. As the regulatory control of foods, feeds, drugs, cosmetics, and pesticides became a recognized governmental function, the AOAC has accepted the responsibility of providing accurate methods of analysis. These standard methods are used not only in enforcement of laws but by both industry and government as a basis for specifications and contracts, and also by research workers in agriculture. The value and significance of research work which includes data on the protein fat, and carbohydrate composition of food, for example, depends directly on standard methods for determining each of these items.

In this 9th edition (1960) the AOAC presents its methods as revised since the publication of the 8th edition in 1955. New chapters on both disinfectants and drugs in feeds have been added. Numerous methods have been added in other chapters, especially in the fields of fertilizers, pesticides and pesticide residues, beverages, flavors, drugs, and nutritional adjuncts. The chapter on soils found in previous editions has been omitted.

Chromatographic techniques have been applied in nearly every field of methods development. Many new methods consist of separating the desired component by chromatography and then determining it colorimetrically. A few paper chromatographic procedures are included for the first time. For many physiologically active materials, bioassay remains the only practicable method of analysis.

As new methods are constantly being adopted, the number of pages has increased with each edition. The size of the book has been controlled by abbreviating words and omitting articles and prepositions. The format has now been revised to a larger page size containing two columns of type.

The teacher of analytical chemistry should find this volume quite valuable as a reference and for special assignments. The student may have some difficulty in following procedures since they are, of necessity, written as brief as possible.

L. R. STONE
Hess & Clark, Inc.
Ashland, Ohio

Wave Mechanics and Valency

J. W. Linnett, Queen's College, Oxford, England. John Wiley & Sons Inc., New York, 1960. xii + 184 pp. Figs. and tables. 13 × 19 cm. $3.

"The object of this book is to try to explain to the experimental chemist the processes and techniques . . . involved in the application of wave mechanics to the electronic structure of atoms and molecules." The book assumes that the reader has a working knowledge of calculus, such as required of our B.S. chemists, but requires no mathematics beyond this.

In the opinion of this reviewer, the book succeeds eminently in its avowed purpose. True, many an experimentalist will lay the book aside early, complaining that it contains too much mathematics, but people who take this attitude must then restrict themselves to the purely qualitative picture, which involves "undoubtedly great dangers in the employment of purely qualitative or pictorial wave mechanics." The book is not intended, and does not give an exhaustive treatment of the subject; however, it should serve admirably to provide the senior, the beginning graduate student, or the experimentalist a few years out of school, with an introduction to the subject.

Following an introductory chapter which aims to derive or justify the Schrödinger equation in terms which do not too much appeal to this reviewer, three chapters deal with problems which can be solved exactly: the particle in the box, the rotator, and the hydrogen atom. Next follows an introduction to approximation methods, variation and perturbation theory, and its use in the treatment of the helium atom. There follows the treatment of the hydrogen molecule and the hydrogen molecule ion. In a chapter on polyelectronic atoms and diatomic molecules, the subject of hybridization is covered along very conventional lines. The chapter on organic polyatomic molecules is restricted to π electron theory. A final chapter deals with inorganic polyatomic molecules, and treats a variety of different types in a very qualitative manner. Four appendices on particular mathematical points do not add too much to the usefulness of the book.

There is no question that the author has faced a tremendous problem, attempting to compress a vast variety of material into 174 small pages. Considering these problems he has done an excellent job; however, the reader will probably, after completing this little volume, look for more extensive treatment, particularly of polyatomic molecules.

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Comparative Biochemistry of Photoreactive Systems

Edited by *Mary Belle Allen*, Kaiser Foundation Research Institute, Richmond, California. Volume 1 of the Symposia on Comparative Biology of the Kaiser Foundation Research Institute. Academic Press, Inc., New York, 1960. xii + 437 pp. Figs. and tables. 16 × 23.5 cm. $12.

This book deals with a number of aspects of the biochemistry of pigments and the living systems in which they are found. The greater number of the articles discuss plant pigments and plant systems. Emphasis is placed on photosynthesis and the biochemical function, biosynthesis, distribution and chemistry of chlorophyll, carotenoids, and other photosynthetic pigments. Other subjects discussed include phototaxis, photoperiodism, photosensitivity of sea urchins, and of sea anemones, fossil pigments, and pteridine pigments. Vision is only briefly mentioned in the articles on pigments of plant origin in animal phyla and on comparative studies of photoreceptors.

There are some 26 articles in all, mostly in the nature of short reviews. These were first presented at the First Annual Symposium on Comparative Biology of the Kaiser Foundation Research Institute. Discussion following the papers is included.

The objective of presenting a useful collection of recent advances in this field seems to have been achieved. This success stems no doubt from an excellent selection of contributors and from the encouragement of the authors to "present an integrated treatment of their work and its relation to other developments in the field." The writing is generally good, and many of the participants have presented their latest hypotheses and conclusions as well as recent research results.

One of the most exciting new developments in the continuing struggle to uncover the mechanism of the primary light reaction in photosynthesis is the discovery of the apparent cooperation of different wavelengths of visible light in the primary processes. This cooperation can be studied in several ways, and appears to be related to the absorption of light by more than one plant pigment in a single plant. These phenomena are discussed from several points of view by Haxo, by French and co-workers, and by Blinks. Vishniac reports his experiments on a possible chemical role of chlorophyll in photosynthesis, and Kamen makes a strong case for the involvement of hematin compounds in the early stages of energy conversion during photosynthesis—a point of view which seems to be gaining in popularity despite opposition.

Printing, formulas, tables, and figures are satisfactory for the most part, though occasionally lettering on figures has been reduced to a size requiring microscopic examination. There is an author index and a 15-page subject index.

As a reference, this book has most value to the research worker studying photosynthesis or comparative plant biochemistry, but it will also be of use to plant biochemists, plant physiologists, botanists, biochemists, and chemists with interests in photochemistry and pigments.

J. A. Bassham
University of California
Berkeley

Physical Methods of Organic Chemistry. Part 3

Edited by *Arnold Weissberger*, Eastman Kodak Company, Rochester, New York. 3rd revised ed. Interscience Publishers, Inc., New York, 1960. xii + 835 pp. 16 × 23.5 cm. Figs. and tables. $24.50.

Parts I and II have already been reviewed in This Journal, **37,** 552 (1960) and **38,** 222 (1961), respectively. This book contains the following sections: XXVIII. Spectroscopy and Spectrophotometry in the Visible and Ultraviolet, by W. West; XXIX.* Infrared Spectroscopy, by D. H. Anderson,* N. B. Woodall,* and W. West; XXX. Colorimetry and Photometric Analysis, by W. West; XXX. Colorimetry and Photometric Analysis, by W. West; XXXI.* Determination of Fluorescence and Phosphorescence, by N. Wotherspoon* and Gerald Oster;* XXXII.* Light Scattering, by Gerald Oster;* XXXIII. Polarimetry, by W. Heller and D. D. Fitts;* XXXIV.* Optical Rotatory Dispersion, by W. Klyne and A. C. Parker; XXXV. Streaming Birefringence, by H. A. Scheraga* and R. Signer; XXVI.* The Kerr Effect, by Catherine G. Le Fevre and R. J. W. Le Fevre; XXVII.* Determination of the Faraday Effect, by C. E. Waring and R. L. Custer; XXXVIII. Measurement of Dielectric Constant and Loss, by J. G. Powles and C. P. Smyth; XXXIX. Determination of Dipole Moments, by C. P. Smyth.

The asterisked sections are new in this edition. The new authors are also asterisked. It is clear that a major part of this edition of Volume I, Part III, has been rewritten, or is new. The authors have done well.

J. A. Campbell
Harvey Mudd College
Claremont, California

Physical Methods of Organic Chemistry. Part 4

Edited by *Arnold Weissberger*, Eastman Kodak Company, Rochester, New York. 3rd revised ed. Interscience Publishers, Inc., New York, 1960. xii + 969 pp. Figs. and tables. 16 × 23.5 cm. $26.

Parts I, II, and III have already been reviewed in This Journal **37,** 552 (1960), **38,** 222 (1961) and above. Part IV contains the following sections: XL. and Microwave Spectroscopy, by B. P. Dailey; XLI.* Nuclear Magnetic Resonance, by H. S. Gutowsky; XLII.* Paramagnetic Resonance Absorption, by George K. Fraenkel; XLIII. Determination of Magnetic Susceptibility, by P. W. Selwood;* XLIV. Potentiometry, by C. Tanford* and S. Wawzonek;* XLV. Conductometry, by T. Shedlovsky; XLVI.* Determination of Transference Numbers, by M. Spiro; XLVII. Electrophoresis, by D. H. Moore; XLVIII. Polarography, by O. H. Muller; XLIX.* Controlled-Potential Electrolysis, by Louis Meites; L. Determination of Radioactivity, by Bert M. Tolbert* and William E. Siri*; LI. Mass Spectrometry, by David W. Stewart.

An index to the whole of Volume I is included. The asterisked chapters are new in this edition. The new authors are also asterisked. It is clear that this Part IV, Volume I, has been considerably expanded, and very much rewritten. The contributors and editor Weissberger are to be congratulated on the overall excellence of the presentations.

J. A. Campbell
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(*Continued on page A548*)

CHEMISTRY TEXTS FROM PRENTICE-HALL

Inorganic Chemistry of Qualitative Analysis

by ALAN F. CLIFFORD, *Purdue University*

Uses the relationships of qualitative analysis to teach the chemistry of the periodic table in a systematic fashion, showing how the analytical scheme is built on and reflects the systematics of the periodic table. Thus the "solubility rules" are expanded and shown to be examples of trends within the periodic table.

1961 515 pages Text list: $6.95

Organic Chemistry

by KEITH M. SEYMOUR, *Butler University*

Presents those facts and principles which serve to give students a working knowledge of the more important types of organic compounds. Pertinent theories and new concepts are used only after the experimental basis for them has been studied and when they contribute to the understanding of the material under consideration.

1961 321 pages Text list: $6.75

Experimental Nuclear Chemistry

by GREGORY R. CHOPPIN, *Florida State University*

Designed to help teach the techniques and necessary basic theory for utilization of radioisotopes in research and experimentation. A variety of experiments and techniques are included with a discussion of the theory behind each.

1961 256 pages Text list: $6.95

Organic Chemistry, 3rd

by RAY Q. BREWSTER and WILLIAM E. MCEWEN, *University of Kansas*

Offers a thorough, well balanced presentation of organic chemistry with emphasis upon theory and mechanism of reactions. Material is presented in sequential fashion, with each chapter building upon its predecessor.

1961 864 pages Text list: $10.00

Synthetic Inorganic Chemistry

by WILLIAM L. JOLLY, *University of California, Berkeley*

Shows how the synthetic inorganic chemist thinks. The text serves the dual purpose of supplementing lecture material and serving as a laboratory manual for a course in preparative inorganic chemistry.

1960 196 pages Text list: $6.00

Elementary Organic Chemistry

by ERNEST E. CAMPAIGNE, *Indiana University*

Contains an integrated, complete survey of elementary organic chemistry in the initial chapters. Following are ten chapters devoted to hydrocarbons, halogen compounds, oxygen compounds and nitrogen compounds of carbons. The remaining chapters cover the organic chemistry of naturally occurring compounds.

1961 360 pages Text list: $7.50

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BOOK REVIEWS

Introduction to Ceramics

W. D. Kingery, Massachusetts Institute of Technology, Cambridge. John Wiley & Sons, Inc., New York, 1960. xvii + 781 pp. Figs. and tables. 16 × 23 cm. $15.

Those instructors in ceramic engineering who have spent endless hours collecting material for courses which this text is designed to serve will wish that it had been available ten or even five years ago. "Introduction to Modern Ceramics," or "A Survey of Modern Ceramics" are better titles, however, since the title used might be interpreted as having to do with "traditional" ceramics.

This book is divided into five parts and 20 chapters. Part I, Ceramics Today, comments on ceramics in general and Part II, Ceramic Processes, deals with raw materials and forming processes. Part III, Characteristics of Ceramic Solids, and Part IV, Development of Microstructure in Ceramics, are pointed toward students who have some familiarity with such subjects as crystal structure and phase diagrams. Part V is designed for graduates.

The amount of preparation in fundamental sciences is a better basis than traditional class standing for determining the students that should profit from a study of this text. A minimum preparation should include calculus, physics, and physical chemistry, but the maximum appreciation, understanding, and response should be expected of graduate students who have had a thorough training in the fundamentals of traditional ceramics and more than the minimum of the fundamentals mentioned above.

So much material is available for a treatise of this type—the only one of its type—that an author must make some difficult choices in his selections to keep the number of pages within reasonable bounds. But, even though this is a treatment in breadth the following additions are suggested: (1) A complete chapter on the colloidal and rheological properties of the plastic minerals; (2) a complete chapter on polymorphism and phase transformations; (3) an extension of Chapter 7, Surfaces and Interfaces, (4) a discussion of tristimulus colorimetry, as used in ceramics, and luminescence should be added to the chapter on Optical Properties.

The physical make-up, organization, and technical presentation are excellent. The illustrations, especially the drawings, are clear cut and provide a lot of information. An innovation in the binary phase diagrams is the shading of the two-phase areas. Few direct references are given but several are listed at the end of each chapter as suggested reading. Absence of footnotes relieves the reader of this distraction.

To all who wish to become familiar with modern ceramics in a minimum of time, whether they be students, teachers, or engineers in the field, this book is recommended, but this reviewer cannot agree with the implication of the author that the empirical approach in ceramic technology is no longer needed. Ceramics as a science has enjoyed remarkable advances during the last 20 years and this book reflects these advances, but the empirical approach and the necessary judgment that goes with it, is not out-dated.

R. M. KING
Ohio State University
Columbus

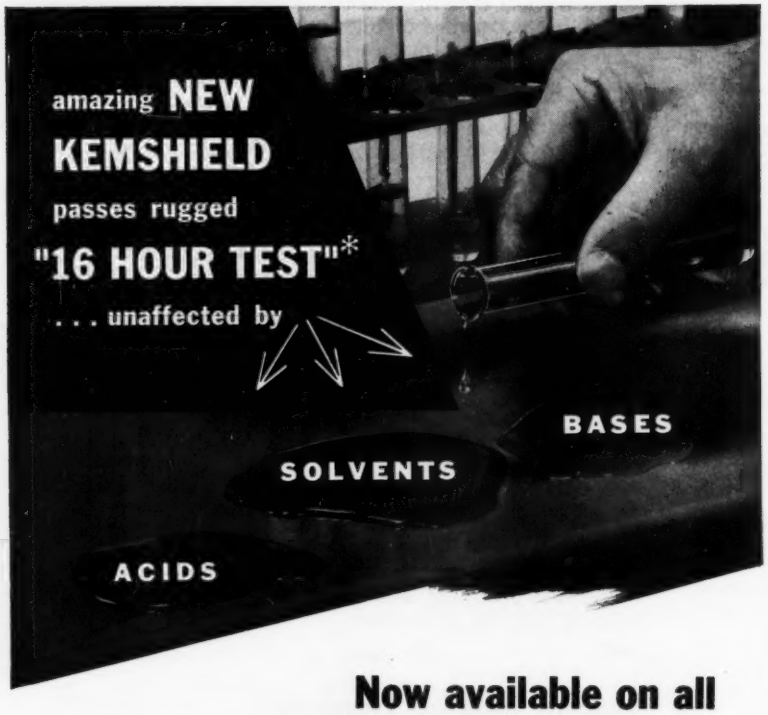
The Surface Chemistry of Metals and Semiconductors

Edited by *Harry C. Gatos*, Lincoln Laboratory, Massachusetts Institute of Technology, Lexington, Massachusetts. John Wiley & Sons, Inc., New York, 1960. xi + 526 pp. Figs. and tables. 15.5 × 23.5 cm. $12.50.

This 521 page book contains the papers presented at the Joint Symposium of the Corrosion and Electronics Divisions of the Electrochemical Society held in Columbus, Ohio, on October 19–21, 1959. The Symposium was jointly sponsored by the Office of Naval Research and Electrochemical Society.

The material presented is divided into

(*Continued on poge A550*)



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By S. YOUNG TYREE, University of North Carolina, and KERRO KNOX, Bell Telephone Laboratories, Inc.

1961, 434 pages, Ill., appendices, $7.00

Designed expressly for the increasing number of undergraduate courses that require no background in physical chemistry, this text surveys the field as a whole while highlighting behavior patterns among the elements.

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789 pages, Ill., $8.50

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5 parts: (I) Chemistry and Physics of Surfaces (4 papers); (II) Imperfections and Surface Behavior (5); (III) Electrode Behavior of Metals and Semiconductors (5); (IV) Surface Reactions in Liquid Media (4); (V) Surface Reactions in Gaseous Media (4). The author of each article is a recognized authority on the subject about which he has written. Eighteen authors were from the U.S., 4 from Germany and 1 each from France and Australia. All papers are uniformly presented in the following form: Abstract-paper-references-discussion. The references given after each paper allow, in most cases, further study of the subject.

The physical and chemical behavior of metal surfaces have been studied for a much longer period than semiconductor surfaces. It is characteristic of the book to compare findings in the metal surface field with the corresponding findings in the semiconductor field. Almost all authors of the papers are from universities or research institutes. The subject of most papers is therefore highly theoretical. For those who want to understand modern theories on surface chemistry the book will be a rich source of information. Paper quality, printing, and the reproduction of illustrations are excellent.

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Mental Drugs: Chemistry's Challenge to Psychotherapy

O. A. Battista, American Viscose Corporation. Chilton Co., Philadelphia. Pennsylvania, 1960. xviii +155 pp. 14.5 × 21 cm. $3.95.

This small volume covers an amazing amount of material in the area of the relationship of chemistry to mental health. Thus such diverse topics as Chemistry of Moods and Emotions, Diet and Mental Health, Chemistry of Mental Retardation, Man-Made Tranquilizers, etc., are treated in an entertaining and informative way. Such esoteric topics as Heath's taraxein, Hofmann's LSD, and Osmond's adrenochrome are introduced to the reader.

It is a bit difficult to decide the type of reader for whom this book was intended. If intended for the chemist, the use of structural formulas for the compounds mentioned would have been helpful. If intended for the clinician, the treatment is somewhat shallow from a medical point of view. In either case the provision of a bibliography would have been helpful to the serious reader.

We may conclude that this book was meant to introduce this subject to the scientist who is not a specialist. In this, the book does a good job—but again a guide to further reading would have been helpful. We are not aware of any other volumes that cover the same ground.

The title is somewhat misleading in that chemical treatment of mental disease is described in the book as an aid to, rather than a replacement for, psychotherapy. Finally, it is our opinion that the lay reader would not benefit greatly from this book. Indeed he may find its oversimplifications somewhat confusing.

MAXWELL GORDON
Smith Kline & French Laboratories
Philadelphia, Pennsylvania

Introduction a l'Etude des Parfums

T. Bassiri. Masson & Cie, Paris, France, 1960. 278 pp. Figs. and tables. 16 × 24 cm. 38NF.

In the preface the author describes his purpose in this work as the preparation of a handbook which will permit the neophyte perfumer "to risk his first steps (in perfumery) without too many errors." In the reviewer's opinion the author has succeeded in compiling a surprisingly large amount of useful and diversified information on perfumery in one compact volume. It will be recognized that any attempt to include in 261 pages of text such diverse topics as (*a*) theories of odor and taste, (*b*) properties of aerosol propellants, (*c*) properties and characteristics of the essential oils, and (*d*) the scientific preparation and attitude of a perfumer will necessitate a rather superficial treatment of any given topic. If we choose to accept this limitation, the author has accomplished his purpose admirably; in so doing he has created a work of very little use to anyone except an apprentice perfumer as any given topic is much more

(*Continued on page A552*)

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☐ **WRITING GUIDE FOR CHEMISTS: Practical Suggestions for Preparing Reports, Dissertations, Articles**

By WALTER J. GENSLER, Boston University; and KINERETH D. GENSLER. Available September, 1961.

A practical guidebook, succinct and example-crammed, to aid chemists, academic, industrial or governmental, in writing reports, dissertations or articles. Part I is a general treatment of style, and traces a report from the laboratory notebook to completion. Part II lists important "do's" and "don't's."

☐ **PHYSICAL CHEMISTRY**

By GORDON M. BARROW, Case Institute of Technology. 720 pages, $8.95.

A modern text aimed at unifying the content of physical chemistry by interpreting quantitative chemical behavior in terms of molecular behavior. All physical chemistry is treated structurally emphasizing the molecular knowledge that can be gained by theory or experiment and applying it to understanding macroscopic behavior. A problem book will be available.

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adequately covered in the standard works of the most modest perfume laboratory.

The text is divided into five sections beginning with a discussion of perfumery as a profession, including a section describing the scientific background (chemistry, physics, botany, cosmetology) necessary to a perfumer. The second section deals with explanations of odor (31 pp.) and taste (6 pp.); many of the important theories relating to each are presented sketchily and uncritically. This is the only section of the book which gives even a minimum number of references to the original literature.

The middle section (74 pp.) discusses perfumes and includes definitions, preparations and 38 formulations which rely heavily on the natural oils rather than synthetics. This section contains much information of value on specialized topics as: perfumes for cosmetics and soaps, aerosol perfumes, and perfumes for plastics.

The final two sections are respectively tabulations of essential oils and the main chemical products used in perfumery. The former section lists 85 of the more common essential oils and includes their botanical derivation, habit, regions of culture, method and yield of isolation, physical constants, constituents and odor. All of this information is available in Gunther in much more detail. The last section contains several hundred entries of "aroma" products (arranged by functional groups) giving for each physical constants, methods of synthesis and areas of application in perfumery. This would appear to be a useful section for first information about the areas of perfumery in which a compound has found application.

The book is an admirable compilation of information; however its diversity severely limits its usefulness in any purpose other than that for which it is written. The French is clear and should present no great obstacles to readers with only a modest command of the language. The format is attractive, and the number of typographical errors is small.

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Inorganic Syntheses. Volume 6

Edited by *Eugene G. Rochow*, Harvard University, Cambridge, Massachusetts. McGraw-Hill Book Co., Inc., New York, 1960. xi + 272 pp. Figs. and tables. 16 × 23.5 cm. $7.75.

The latest volume in this series maintains the high standards set by its predecessors. Detailed procedures for the preparation of more than 96 inorganic substances are given in the 69 entries. The growing international status of this relatively new (1939) publication is reflected by the fact that roughly one-third of the contributions may be credited to foreign chemists, many of whom are world-known authorities.

All the procedures have been checked by at least one independent laboratory, and in many cases improvements of the checkers have been incorporated. Each synthesis contains a critical summary of preparative methods, a procedure including crucial details, a description of properties, both physical and chemical, and representative references. In numerous cases, equations, diagrams, yields, safety precautions, and analytical methods and data are provided. Many of the syntheses are new, while others are improvements and adaptations of existing methods. Occasionally, alternate methods allow comparisons and add to the flexibility of the volume. As has been customary, the book is divided into chapters according to the periodic table, subdivided into A and B subgroups. The usual cross-references, an index of contributors and checkers, and cumulative subject and formula indexes are provided.

Manuals for courses in inorganic preparations are still scarce; although intended primarily for the research chemist, this volume will provide the instructor of such a course with a gold mine of material for use in the college laboratory. Among the wide variety of techniques and reactions used are the following: nonaqueous solvents, arc-induced and amalgam reductions, inert or anhydrous atmospheres, and preparation of deuterium-labeled acids. The types of preparations illustrated range from simple to complex compounds of both common and rare elements and include anhydrous halides and oxyhalides, mononuclear and polynuclear complexes, N-P

(*Continued on page A554*)

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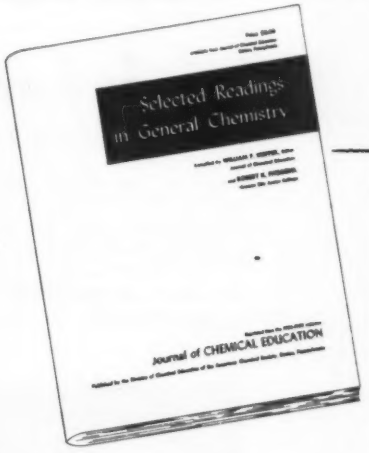
Compiled by **WILLIAM F. KIEFFER** and **ROBERT K. FITZGEREL**



"Selected Readings in General Chemistry," is a 128-page collection of over 40 papers which have appeared in dozens of separate issues of the *Journal of Chemical Education* during the last three years. On the list of authors, such names as Calvin, Libby, Nyholm, Pauling, and Williams are recognized by chemists everywhere. Topics range from spontaneous generation of life to solid state models, and from antiknock quality in gasoline out into space with the IGY and amateur rocketry.

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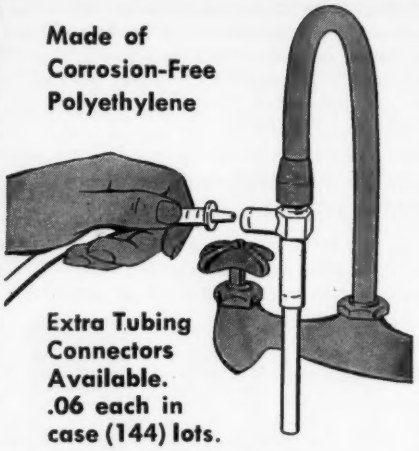

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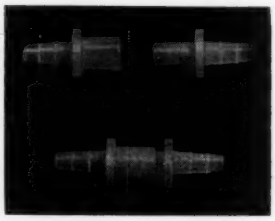

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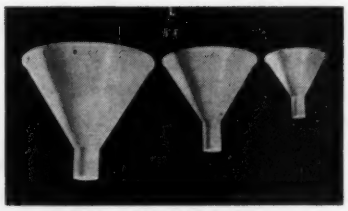

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BOOK REVIEWS

and N-S polymers, and π-bonded sandwich compounds. Unusual oxidation states are well represented. Of late, much inorganic research has involved borderline inorganic-organic compounds. Accordingly, preparations utilizing the Grignard, diazotization, and other reactions long familiar to the organic chemist are given.

The series has been criticized for the lack of balance in subject matter, a result of the editorial policy of assembling each volume from unsolicited manuscripts. Such a policy, which in this volume produces a heavy emphasis on phosphorus compounds and coordination complexes, especially biguanide complexes, does, however, have the advantage of reflecting current research trends. In view of the diversity of contributors, the Editorial Board is to be commended for producing a uniform volume relatively free of errors. Because it has been repeatedly emphasized that *Inorganic Syntheses* is a journal rather than a book and in view of the usefulness of the procedures to both practicing chemists and chemical educators, the publishers should seriously consider making provision for reprints.

GEORGE B. KAUFFMAN
Fresno State College
Fresno California

College Chemistry: A Systematic Approach

Harry H. Sisler, University of Florida, Gainesville, and *Calvin A. VanderWerf* and *Arthur W. Davidson*, both of the University of Kansas, Lawrence. 2nd ed. The Macmillan Co., New York, 1961. x + 709 pp. Figs. and tables. 16 × 23.5 cm. $7.50.

The outstanding features of the book are: Clarity of presentation; an emphasis on the fundamental concepts of chemistry with the structural basis of the properties of matter serving as the unifying principle; the student is made aware of the "unfinished business" of chemistry with challenges and problems; the needs of students of various capabilities are served with quantitative problems of graduated difficulty.

The book is up-to-date. Molecular orbital concepts are considered and chapters on "The Chemistry of Life," "The Realm of the Giant Molecule," and "Chemistry Rockets and Outer Space" are presented.

The principles of qualitative analysis are not introduced.

In the opinion of the reviewer, this text exhibits a basic deficiency that is also found in other general chemistry texts: the fundamental principles and concepts presented in the first portion of the book are not sufficiently correlated with the descriptive chemistry of the latter half of the book.

PAUL H. CARNELL,
Albion College,
Albion, Michigan

a Chem Ed feature

CHEMICAL PROJECTS

Research Ideas for Young Chemists

Compiled by **JAY A. YOUNG,** Kings College, Wilkes-Barre, Penna.
JOHN K. TAYLOR, National Bureau of Standards, Washington, D. C.

Coulometric Titrations

See REILLEY, C. N., *J. Chem. Educ.*, **31**, 543–545 (1954).

In a typical titration a solution of one reagent is added from a buret to the solution of another in a beaker or flask. In a coulometric titration, the added reagent is generated within the liquid in the flask by an electrolytic process. The current is held constant at a known value, and the amount of reagent generated is determined from a knowledge of the electrolytic reaction and the total elapsed time.

The constant current source can be constructed in a few hours from electronic components available at radio-part stores. An accurate timer is recommended, but an ordinary electric clock with a sweep second hand can be substituted (an obvious modification of the wiring in the circuit diagram, Figure 2 of the article, is necessary). For precise work, the error introduced by the slow start and prolonged, coasting stop of such a clock can be determined by the coulometric titration of a solution of known composition.

Details pertaining to the coulometric titration of arsenite by electrolytically generated iodine are described in the article as an example. Other titrations are possible.

Questions:

Determine the percentage of arsenic present in an arsenical insecticide.

Devise a suitable analytical procedure, using electrolytically generated chlorine, for the analysis of a substance of your own choosing.

Coulometric titrations are suitable for the analysis of very small quantities of sample material. What is the smallest weight of sample for which, in comparison with a volumetric analysis of the same material, results of equal precision can be obtained?

Devise your own coulometric titration procedure for the analysis of a substance which you have selected.

The discussion in the article indicates that coulometric titration can be used in place of titration by buret, when the buret contains a solution of an oxidizing agent. Can coulometric titration also be used in place of a titration by buret if the buret were to contain a solution of a reducing agent?

Can coulometric titration be used for the analysis of acids or bases?

The Senses as Indicators in Volumetric Analysis

See PHILLIPS, J. P., *J. Chem. Educ.*, **35**, 35 (1958).

In addition to a change of color, other changes could be used to indicate the endpoint of a titration. For example, the taste of a strong acid, the production of an odorous compound, the loss of lubricity of a basic solution, and other similar effects have been used as the basis of an endpoint indication. References describing these details are cited.

Because of their hazardous nature, explosometric titrations to an auditory endpoint should not be studied by a beginner.

Question:

Determine a method for indicating the endpoint of a titration other than the change in color of an indicator. A satisfactory method should be approximately as sensitive as the common color-change indicator systems and yield reproducible results in the hands of any trained chemist. It must, of course, be free from hazard. Evaluate the method you devise by these criteria.

Quantitative Results of Volumetric Analysis—A Graphic Procedure

See TRETSVEN, W. I., *J. Chem. Educ.*, **24**, 565 and 572 (1947).

In all but the most unusual titrations, the volume of titrant used to reach the endpoint is proportional to the quantity of reagent originally present in the unknown solution, within almost negligible error. Hence, it is possible to determine the quantity of unknown present by graphical means, using linear plots; and this mode of calculation is advantageous when several, repetitive, determinations must be carried out. As an example, this article describes the determination of the amount of salt present in a weighed sample of cheese, but the method can be adapted to almost any volumetric determination.

Questions:

Select any volumetric determination you please which can be carried out either directly or by back titration of an excess of added reagent. Set up a graphical description of the selected procedure. Using solutions of unknown concentration, carry out several determinations of the concentration of the unknown and compare the precision of the results you obtain from the graphical computation with the precision of the results obtained by ordinary arithmetic calculations.

Which method for determining the concentration of an unknown is to be preferred?

Can the precision obtainable from the graphical computation be improved by plotting the data on a larger scale?

The accuracy (not the precision) of many titrations can be improved by including a correction for a blank titration. Tretsven states that this kind of correction can be incorporated into a graphical computation. Select a titration for which a blank correction should, properly, be made; verify or disprove Tretsven's statement.

Separation of Organic Compounds with Urea and Thiourea

See KOBE, K. A., AND REINHART, L. R., *J. Chem. Educ.*, **36**, 300–1 (1959).

Urea, thiourea, and many other compounds can form unusual crystalline complexes with several different substances. For example, thiourea molecules will form in a hexagonal helix around carbon tetrachloride, thus entrapping it, and the crystals which result can be separated from the liquid milieu by filtration. When water is added to the crystals, the thiourea dissolves and the carbon tetrachloride is released, forming a separate layer. In this manner carbon tetrachloride can be separated from octanol since octanol does not form a similar complex with thiourea.

The formation of an adduct, or clathrate compound, can be predicted from a knowledge of the dimensions of the canal in a helical configuration of the host compound and of the size of the guest molecule. A list of seven compounds that form clathrates with urea, and of seven others that form clathrates with thiourea, is given; this list is only suggestive since many other guests are known and several other compounds are known to be hosts. Iodine, for example, forms a clathrate compound with starch helices. The use of starch as an indicator in iodometric titrations depends upon this phenomenon since the iodine-starch clathrate compound is blue in color.

Questions:

In almost every analytical problem, the substance being analyzed must be separated from other substances present and then, once separated, its amount is determined in a following step. If the separation is incomplete, or if other substances are also separated, the final analysis will be in error. Since their recent discovery, clathrate compounds have provided a new tool for the analytical chemist to use in separation procedures. Carry out a suitable synthesis of an organic compound and determine the yield of product formed by separating the desired product formed as a clathrate compound.

Determine the composition of a sample of kerosene or of gasoline.

Show that two optical isomers can be separated by the formation of clathrate compounds.

Acidimetric Determination of Metal Ions

See ALTSCHUL, R., AND ALTMAN, H., *J. Chem. Educ.*, **27**, 278–80 (1950).

When a metal sulfide is precipitated from aqueous solution by the addition of hydrogen sulfide, an equivalent quantity of hydronium ion is also formed.

$$2H_2O + M^{++} + H_2S \rightleftarrows MS + 2H_3O^+$$

If the metal sulfide is sufficiently insoluble, titration of the acidic solution that results can be used to determine the quantity of metal ion originally present. If the sulfide is slightly soluble, however, this determination will not work. In some cases, with Ni^{++}, for example, NiS precipitates slowly as the mixture is titrated, and, with patience, good results can be obtained. When the color of the sulfide that is formed does not interfere with the color of the indicator that is chosen, the endpoint can be detected by a change in the color of the indicator; usually; however, it is better to plot a titration curve, using a *p*H meter.

Questions:

Compare the results obtained from a more common method of analysis with the results obtained from the procedure described by Altschul and Altman, using one or more metals or metal compounds of your own choice.

If their equivalent weights are sufficiently different, the percentage composition of two metals in an alloy or particulate mixture can be determined by the method described here. Why is it necessary that the equivalent weights of the two metals be markedly different, in order to obtain reasonably accurate results?

Separate a mixture of two or more heavy metal salts by using different conventional procedures; compare the efficacy of the procedures you have used by precipitating the separated metals as sulfides, in a final step, and titrating the hydronium ion that is formed.

Suggest an explanation for any differences in the efficacy of the separation procedures you have used and test your explanation by further laboratory work.

Manganese(II) ion cannot be determined quantitatively by the method of Altschul and Altman because MnS is soluble in mildly acidic solutions. Can you devise a modified procedure for the quantitative determination of manganese, based on the method described here?

Other substances, such as the hydrogen form of an ion exchange resin, can be used in place of H_2S in an otherwise similar quantitative determination. Select one such suitable substance and devise a workable quantitative analytical procedure for one or more metals of your own choice.

Preparation of 2,4,6-tri-t-butylphenol

See SOMERS, B. G., AND COOK, C. D., *J. Chem. Educ.*, **32**, 312–13 (1955).

As an example of a Friedel-Crafts alkylation, the preparation of this phenol derivative is suggested. The reaction goes smoothly, requiring the controlled addition of a gas, isobutylene, to a solution of phenol in benzene in the presence of sulfuric acid at a moderately high temperature. When oxidized, the product forms a free radical which is reasonably stable and of historical interest.

Questions:

Prepare 2,4,6-tri-t-butylphenol by the method here described and determine the conditions which affect the yield. Can you identify the by-products also formed? What are some of the chemical properties of 2,4,6-tri-t-butylphenol?

Can you prepare another compound by the same procedure, using a starting material other than phenol?

Can you prepare another compound by the same procedure, using a gas or volatile liquid other than isobutylene?

Compare the properties of 2,4,6-tri-t-butylphenol with those of phenol or of other phenol derivatives. What are the reasons for the differences in properties? Devise suitable laboratory procedures which will test the validity of your explanations of these differences.

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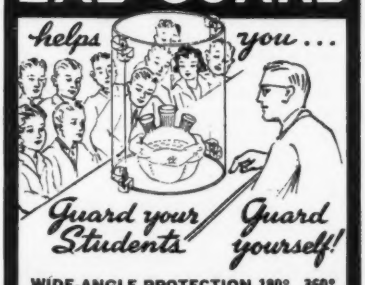

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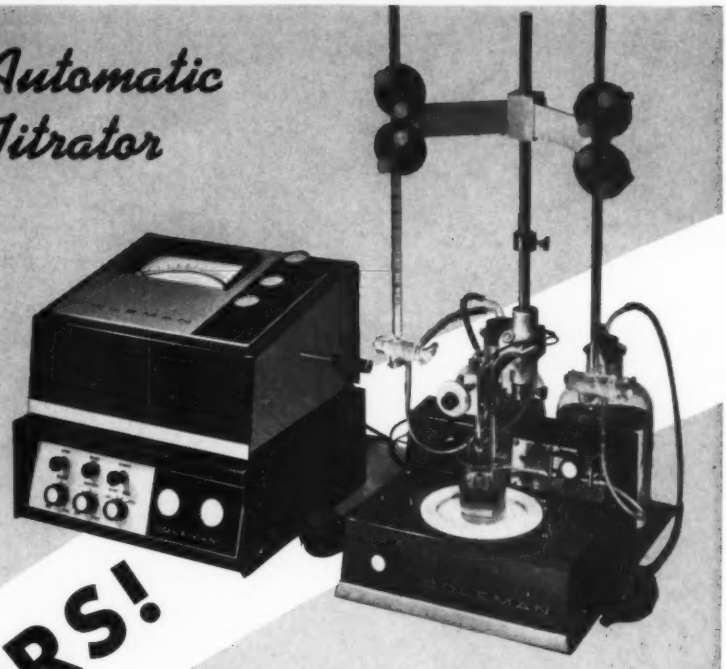

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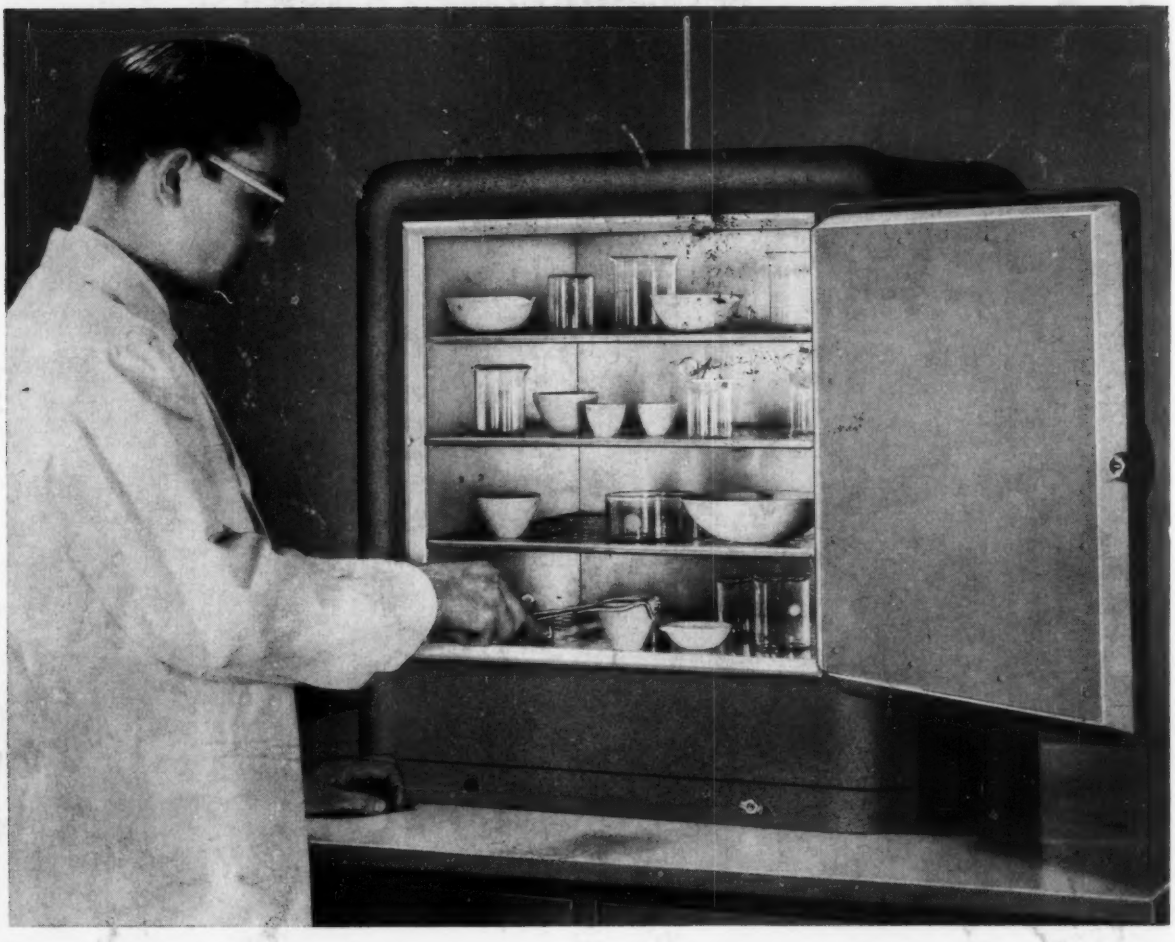

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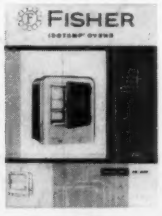

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